

**NARRATIVE**

December 16, 1992

Narrative Project: 92-321  
Reference No.: 32359-51  
Client: WHC  
SDG No.: 3410

**VOLATILES**

The samples were analyzed according to the OLM01.8 Statement of Work. The samples in this SDG were very clean and the analyses non-problematic. Only Methylene Chloride and Acetone were detected in concentrations similar to those found in the blank.

The quality control results were acceptable. All surrogate recoveries were excellent. There was no MS/MSD set for this SDG. The LCS was found to have severe interference from the sample analyzed prior to it, and consequently it is not reported. All samples were analyzed within holding time and the blank was clean. All initial and continuing calibration data are compliant.

**SEMIVOLATILES**

The samples were analyzed according to the OLM01.8 Statement of Work. The sample was very clean and the analyses non-problematic. Only target analyte detected was Di-n-butylphthalate (260 ppb). DDT was detected as a TIC.

The quality control results were generally acceptable. Surrogate recoveries were within QC limits. All LCS recoveries were excellent. Please note that Di-n-Octylphthalate was extra in the matrix spiking solution. The results are reported on form I, flagged with "X", but recovery data are not included on form III. All samples were extracted within holding times and the blanks were clean. All initial and continuing calibration data are compliant.

**ORGANOCHLORINE PESTICIDE/PCBs**

The samples were analyzed according to the OLM01.8 Statement of Work. The sample B07KP6 needed to be analyzed at a 1:10 dilution in order to bring DDT and

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## ***NARRATIVE***

DDE into calibration range. Both undiluted and diluted analyses are reported. DDT concentration in the sample was around 330 ppb, and DDE at 260 ppb. Both were confirmed by GC/MS. Also DDD was found in the GC/MS analysis of the semivolatile fraction. However, it was not found on column DB-1701, although it was found on DB-608. DDD is not reported as detected in the sample, but it is possible that it is in the sample, and the retention time shifted on column DB-1701 for some reason. If this was the case, the estimated concentration of DDD in the sample would be around 100 ppb.

The sample was extracted within holding time, and the blank was free of contamination. All initial and continuing calibration data are compliant.

The quality control results were generally acceptable. All surrogate recoveries are within QC limits for the sample. However, all recoveries were slightly low (51-59%) for the blank, and Tetrachloro-m-xylene recovery is slightly low (59%) for the LCS on column DB-1701 only. All LCS recoveries were excellent. There was no MS/MSD set for this SDG.

### **ORGANOCHLORINE HERBICIDES**

The samples were analyzed according to SW-846 Method 8150. Sample BO7KP6 contained no herbicides.

The quality control results were acceptable. Surrogate recoveries were acceptable. LCS recoveries were excellent.

Please note that the quantitation column was DB1701 and the confirmation column was DB608. Quantitative information is not rigorously reviewed for the confirmation column. Calibration results were acceptable.

### **ORGANOPHOSPHORUS PESTICIDES**

The samples were analyzed according to SW-846 Method 8140. There were no hits in this group of samples.

A 5 point calibration curve was run for individual component pesticides. A 3 point calibration curve was used for some of the problem compounds. Continuing calibration was high on most of the "A" mix compounds, the "B" mix compounds were generally acceptable.

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The quality control results were generally acceptable. The surrogate recoveries were approximately 150% for all compounds. Ethion was the surrogate spike, calibrated from the "B" mix. The high bias is probably due to a standards prep error.

LCS, MS and %RPD recoveries were generally acceptable for several compounds. Problems occurred with M.parath, Merphos, Sulprophos, and Coumaphos which are typically problem compounds.

### **TRPH**

The samples were analyzed according to EPA Method 418.1 for TRPH. Please note that these samples were batched with another group of samples. The quality control results were acceptable.

### **METALS**

The samples were analyzed according to the ILM02.1 Statement of Work for the CLP metals list. The GFAA metals were initially analyzed undiluted. Due to low analytical spike recoveries, the GFAA metals were reanalyzed at a dilution of 10 with acceptable results. Only the diluted samples are reported.

The quality control results were acceptable. Pb was detected in the prep blank greater than CRDL, however, the level in the sample is greater than 10 times the amount in the blank and no corrective action was necessary. The soil LCS results were within advisory ranges.

### **ANIONS**

The samples were analyzed according to EPA Method 300.0 for anion.  $\text{SO}_4$  required a dilution of 10 times due to high concentration level. All other anions were reported on straight analysis run. For soil, 20 gm of sample was leached into 100 ml of DI Type II water prior to analysis. The quality control results were acceptable. MS and %RPD were within the control limits.

### **CHROME IV**

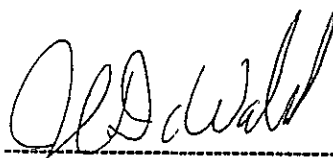
The samples were analyzed according to EPA Method 7196 for colorimetric Chrome VI analysis. The sample required a 1:5 dilution prior to analysis due to matrix interferences. For soil, 20 gm of sample was leached into 100 ml of DI Type II water prior to analysis. The quality control results were acceptable.

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## ***NARRATIVE***

### **NO2/NO3**

The samples were analyzed according to EPA Method 353.3 for NO2/NO3. For soil, 20 gm of sample was leached into 100 ml of DI Type II water prior to analysis. The quality control results were acceptable.



John DeWald  
Project Manager

enclosures

r:\narr\n3410

id 4mm 12-2-94

<b>Westinghouse Hanford Company</b>		<b>NONCONFORMANCE REPORT</b>			1. Page <u>1</u> of <u>1</u>		2. Preprinted No. <b>051181</b>		
							QA Log No. <b>EQA-92-117</b>		
3. P. O., W. O., or Job Control No. <b>N/A</b>		4. System/End Use <b>N/A</b>		5. Item/Material <b>N/A</b>		6. Dwg./Spec./Other No. <b>N/A</b>		7. Rev. <b>N/A</b>	
8. Program/Project/Other <b>North Slope Expedited Response Action</b>					9. Safety Class <b>N/A</b>		10. ASME Code Items <input type="checkbox"/> Yes <input checked="" type="checkbox"/> No (If yes, notify authorized inspector)		
11. Supplier Name/Address <b>Data Chem 960 West Levoy Drive Salt Lake City, UT 84123-2547</b>						12. Notification of Potential Occurrence Required <input type="checkbox"/> Yes <input checked="" type="checkbox"/> No			
13. Code: Lot/Heat/Serial <b>N/A</b>		14. Lot Size <b>N/A</b>		15. Sample <b>N/A</b>		16. Qty. Acc. <b>N/A</b>		17. Inspection Criteria <input type="checkbox"/> Dwg. <input type="checkbox"/> Spec. <input type="checkbox"/> Insp. Plan <input checked="" type="checkbox"/> Other <u>WHC-SD-EN-AP-099, Rev.</u>	
18. Item		19. Description of Nonconformance (list serial no. where applicable)				22. Disposition, Justification, and Instructions			
1.		North Slope Expedited Response Action				Accept, lab sample method from data chem.			
		Sampling Plan WHC-SD-EN-AP-099, Rev. 1				Error in WHC Sample Plan, WHC-SD-EN-AP-099			
		specified the phosphorus pesticide				Rev. 1 laboratory method was not correct.			
		analysis method to be SW-846/8190.				Change table 2 page 7 line "Phosphorus			
		The				pesticides" second column to SW-846/8140.			
		actual analytical method is SW-846/8140							
		which the analytical lab utilized in the							
		phosphorus pesticide analysis.							

20. Originator's Signature <i>C. L. Menley</i>		Date <b>12/8/92</b>		23. Design Document Change Required? <input type="checkbox"/> Yes, Occ. No. _____ <input type="checkbox"/> No	
21. Cognizant QA Manager's Signature <i>[Signature]</i>		Date <b>12-22-92</b>		24. Corrective Action Required? <input type="checkbox"/> Yes, No. _____ <input type="checkbox"/> No	
Disp. App.	25. Cognizant Engineer <i>C. L. Menley</i>	Date <b>12/8/92</b>	26. Technical Rep.	Date	Signature/Org.
	QA Engineer <i>[Signature]</i>	Date <b>12/8/92</b>	Signature/Org.	Date	Signature/Org.
Close	27. <input checked="" type="checkbox"/> Accept <input type="checkbox"/> Reject <input type="checkbox"/> Follow on NCR				
	<i>[Signature]</i> QA/C Personnel				<b>12/21/92</b> Date

**DON'T SAY IT --- Write It!**

**DATE:** September 1, 1993

**TO:** File 3410-SCU-080

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**FROM:** Linda J. Dickerson

**H4-19**

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**Telephone:** 372-2895

**cc:** 3395-SCU-078

**SUBJECT:** Validation Summary Final Report

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Final validation report for this package is filed with 3395-SCU-078

Westinghouse Hanford  
Company

CHAIN OF CUSTODY

Custody Form Initiator J. G. Lucas

Company Contact Frank Gustafson

Telephone (509) 376-1736

Project Designation/Sampling Locations North Slope EIA -  
H-06-H(E)

Collection Date 11-2-92

Ice Chest No. RM #5

Field Logbook No. EFL-1031

Bill of Lading/Airbill No. 251900658 9

Offsite Property No. W93-U-000238

Method of Shipment EMERY

Shipped to S-Cube San Diego, CA

Possible Sample Hazards/Remarks —

Sample Identification

B07KPG - (1) 120ml aS

(1) 250 ml aG

(3) 120 ml aG

☐ Field Transfer of Custody

CHAIN OF POSSESSION

(Sign and Print Names)

Relinquished by: Jonathan G. Lucas

Received by:

Date/Time:

Donna L. Lucas

Relinquished by:

Received by:

Date/Time:

Relinquished by:

Received by:

Date/Time:

Relinquished by:

Received by:

Date/Time: 2:00

Frank Gustafson

11-2-92

Final Sample Disposition

Disposal Method:

Disposed by:

Date/Time:

Comments:

100% of sample was analyzed

2:30 PM

12-2-92



11-3-92

Date 11-2-92 (corrected)

Telephone (509) 376-1736

*Type of Sample	A = Air	L = Liquid	SE = Sediment	T = Tissue	X = Other
	DL = Drum Liquids	O = Oil	SL = Sludge	W = Water	
	DS = Drum Solids	S = Soil	SO = Solid	WI = Wipe	

### Possible Sample Hazards



# SDG Memo/Sample Summary



Client Name: WESTINGHOUSE HANFORD CO. Date: 4 Dec 1992  
 Project Name: 92-321 Update No.:  
 SDG No.: 3410 Work Order No.: 32359-51  
 Project Manager: J. DEWALD  
 Mail Date:

Client Samp No.	S-Cubed Samp No.	Date Rcvd	Date Samp	Matrix	ANIONS	CRVI	FURNCLP	HERBEXT	HGCLP	ICPCLP	NO2/NO3	OCPOLM	OPP8140	SVOAOLM	TRPH	VOAOLM
B07KP6	3410-01	11-6-1992	11-2-1992	SOIL	X	X	X	X	X	X	X	X	X	X	X	X

(X) = Non-Billable Sample

1/2 Jan 12-2-94

PLEASE TYPE OR USE BALL POINT PEN. BEAR DOWN FIRMLY!  
KEEP MARKS WITHIN BOXES TO ASSURE ACCURACY

FORM OF PAYMENT				SERVICES			
Check <input type="checkbox"/> ON <input type="checkbox"/> <span style="float: right;">FCCOD <input type="checkbox"/></span> Bill to Shipper <input checked="" type="checkbox"/> Bill to Consignee <input type="checkbox"/> Third Party Billing <input type="checkbox"/>				UNITED STATES (AMERICA) <input type="checkbox"/> Express <input type="checkbox"/> <input type="checkbox"/> Same Day (Extra Charges) <input type="checkbox"/> Standard Plus <input type="checkbox"/> Business Documents <input type="checkbox"/> <input checked="" type="checkbox"/> AM <input type="checkbox"/> PM <input type="checkbox"/> Preferred <input type="checkbox"/> Customs Clearance <input type="checkbox"/> <input type="checkbox"/> Second Day <input type="checkbox"/> Standard <input type="checkbox"/> Delivery <input type="checkbox"/>			
Shipper's Account Number <b>E-850281585</b>				<b>EMERY</b> <b>WORLDWIDE</b> 			
Date <b>11-04-92</b> Origin <b>PSC</b>				2519006589			
From: <b>WESTINGHOUSE SHIPPING DEPT (509) 376-6665</b> <b>U.S. DEPARTMENT OF ENERGY C/O</b> <b>WESTINGHOUSE HANFORD</b> <b>BLDG 1163</b> <b>2355 STEVENS DRIVE</b> <b>RICHLAND WA</b>				To: <b>JOHN DEWALD</b> <b>S-CUBED</b> <b>3398 CARMEL MT. ROAD</b> <b>SAN DIEGO CA</b>			
Customer's Reference Numbers <b>W81300 PD42A W93-0-0002#38</b>				Consignee's Account Number <b>99352</b>			
Description <b>1 ICE CHEST RM#5</b> <b>SOIL SAMPLES</b> <b>B07KP6</b>				Dimensions Pcs <b>1</b> L <b>17</b> W <b>14</b> H <b>17</b> Total Pcs <b>1</b> Total Weight <b>22</b>			
Remarks Shipper's Signature <i>[Signature]</i>				Mark if Emery Packaging is used Urgent Letter <input type="checkbox"/> Urgent Pack <input type="checkbox"/> <b>9X12</b> <input type="checkbox"/> <b>12X15</b> <input type="checkbox"/>			
International Shipments Free Domestic <input type="checkbox"/>				Third Party Account Number <b>E</b>			
Base Charge				International Customs Value International Insurance Total Transportation Charges			
Other Charges/Advice at Origin <input type="checkbox"/> <b>NO</b> \$				Declared Value \$			
FOR INFORMATION OR HATT CALL 1-800 44 EMERY (1-800 443 6379)							
2519006589 							
<b>SAN-A</b> Terms and Conditions on Back							

FULL FOR  
SHIP.  
NO  
TAB

CONSIGNEE - PACKAGE COPY - 4



1A  
VOLATILE ORGANICS ANALYSIS DATA SHEET

EPA SAMPLE NO.

B07KP6

Lab Name: S-CUBED Contract: 32359-51  
Lab Code: S3 Case No.: 92-321 SAS No.: SDG No.: 3410  
Matrix: (soil/water) SOIL Lab Sample ID: 3410-01  
Sample wt/vol: 5.00 (g/ml) G Lab File ID: A1110061  
Level: (low/med) LOW Date Received: 11/05/92  
%Moisture: not dec. 5.67 Date Analyzed: 11/10/92  
GC Column: PACK ID: 2.00 (mm) Dilution Factor: 1.00  
Soil Extract Volume: (uL) Soil Aliquot Volume: (uL)

CAS NO. COMPOUND CONCENTRATION UNITS:  
(ug/L or ug/Kg) ug/kg Q

74-87-3	Chloromethane	11	U
74-83-9	Bromomethane	11	U
75-01-4	Vinyl Chloride	11	U
75-00-3	Chloroethane	11	U
75-09-2	Methylene Chloride	6	Jβ
67-64-1	Acetone	7	Jβ
75-15-0	Carbon Disulfide	11	U
75-35-4	1,1-Dichloroethene	11	U
75-34-3	1,1-Dichloroethane	11	U
540-59-0	1,2-Dichloroethene (total)	11	U
67-66-3	Chloroform	11	U
107-06-2	1,2-Dichloroethane	11	U
78-93-3	2-Butanone	11	U
71-55-6	1,1,1-Trichloroethane	11	U
56-23-5	Carbon Tetrachloride	11	U
75-27-4	Bromodichloromethane	11	U
78-87-5	1,2-Dichloropropane	11	U
10061-01-5	cis-1,3-Dichloropropene	11	U
79-01-6	Trichloroethene	11	U
124-48-1	Dibromochloromethane	11	U
79-00-5	1,1,2-Trichloroethane	11	U
71-43-2	Benzene	11	U
10061-02-6	trans-1,3-Dichloropropene	11	U
75-25-2	Bromoform	11	U
108-10-1	4-Methyl-2-pentanone	11	U
591-78-6	2-Hexanone	11	U
127-18-4	Tetrachloroethene	11	U
79-34-5	1,1,2,2-Tetrachloroethane	11	U
108-88-3	Toluene	11	U
108-90-7	Chlorobenzene	11	U
100-41-4	Ethyl Benzene	11	U
100-42-5	Styrene	11	U
1330-20-7	Xylene (total)	11	U

Sample Number  
BC7KPG

Organics Analysis Data Sheet  
(Page 4)

Tentatively Identified Compounds

CAS Number	Compound Name	Fraction	RT or Scan Number	Estimated Concentration (ug/l or ug/kg)
1.	NO PC'S FOUND	VOA		
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1B  
SEMIVOLATILE ORGANICS ANALYSIS DATA SHEET

EPA SAMPLE NO.

B07KP6

Lab Name: S-CUBED Contract: 32359-51  
Lab Code: S3 Case No.: 92-321 SAS No.: SDG No.: 3410  
Matrix: (soil/water) SOIL Lab Sample ID: 3410-01  
Sample wt/vol: 30 (g/ml) G Lab File ID: DN12061  
Level: (low/med) LOW Date Received: 11/05/92  
%Moisture: 5.67 decanted: (Y/N) N Date Extracted: 11/09/92  
Concentrated Extract Volume: 2000.00 (uL) Date Analyzed: 11/12/92  
Injection Volume: 1.00 (uL) Dilution Factor: 1.00  
GPC Cleanup: (Y/N) Y pH: 8.95

CAS NO. COMPOUND CONCENTRATION UNITS:  
(ug/L or ug/Kg) ug/kg Q

108-95-2	Phenol	700	U
111-44-4	bis(2-Chloroethyl) ether	700	U
95-57-8	2-Chlorophenol	700	U
541-73-1	1,3-Dichlorobenzene	700	U
106-46-7	1,4-Dichlorobenzene	700	U
95-50-1	1,2-Dichlorobenzene	700	U
95-48-7	2-Methylphenol	700	U
108-60-1	2,2'-oxybis(1-Chloropropane)	700	U
106-44-5	4-Methylphenol	700	U
621-64-7	N-Nitroso-di-n-propylamine	700	U
67-72-1	Hexachloroethane	700	U
98-95-3	Nitrobenzene	700	U
78-59-1	Isophorone	700	U
88-75-5	2-Nitrophenol	700	U
105-67-9	2,4-Dimethylphenol	700	U
111-91-1	bis(2-Chloroethoxy) methane	700	U
120-83-2	2,4-Dichlorophenol	700	U
120-82-1	1,2,4-Trichlorobenzene	700	U
91-20-3	Naphthalene	700	U
106-47-8	4-Chloroaniline	700	U
87-68-3	Hexachlorobutadiene	700	U
59-50-7	4-Chloro-3-methylphenol	700	U
91-57-6	2-Methylnaphthalene	700	U
77-47-4	Hexachlorocyclopentadiene	700	U
88-06-2	2,4,6-Trichlorophenol	700	U
95-95-4	2,4,5-Trichlorophenol	1700	U
91-58-7	2-Chloronaphthalene	700	U
88-74-4	2-Nitroaniline	1700	U
131-11-3	Dimethylphthalate	700	U
208-96-8	Acenaphthylene	700	U
606-20-2	2,6-Dinitrotoluene	700	U
99-09-2	3-Nitroaniline	1700	U
83-32-9	Acenaphthene	700	U

1C  
SEMIVOLATILE ORGANICS ANALYSIS DATA SHEET

EPA SAMPLE NO.

B07KP6

Lab Name: S-CUBED Contract: 32359-51  
Lab Code: S3 Case No.: 92-321 SAS No.: SDG No.: 3410  
Matrix: (soil/water) SOIL Lab Sample ID: 3410-01  
Sample wt/vol: 30 (g/ml) G Lab File ID: DN12061  
Level: (low/med) LOW Date Received: 11/05/92  
%Moisture: 5.67 decanted: (Y/N) N Date Extracted: 11/09/92  
Concentrated Extract Volume: 2000.00 (uL) Date Analyzed: 11/12/92  
Injection Volume: 1.00 (uL) Dilution Factor: 1.00  
GPC Cleanup: (Y/N) Y pH: 8.95

CAS NO. COMPOUND CONCENTRATION UNITS:  
(ug/L or ug/Kg) ug/kg Q

51-28-5	2,4-Dinitrophenol	1700	U
100-02-7	4-Nitrophenol	1700	U
132-64-9	Dibenzofuran	700	U
121-14-2	2,4-Dinitrotoluene	700	U
84-66-2	Diethylphthalate	700	U
7005-72-3	4-Chlorophenyl-phenyl ether	700	U
86-73-7	Fluorene	700	U
100-01-6	4-Nitroaniline	1700	U
534-52-1	4,6-Dinitro-2-methylphenol	1700	U
86-30-6	N-Nitrosodiphenylamine (1)	700	U
101-55-3	4-Bromophenyl-phenylether	700	U
118-74-1	Hexachlorobenzene	700	U
87-86-5	Pentachlorophenol	1700	U
85-01-8	Phenanthrene	700	U
120-12-7	Anthracene	700	U
86-74-8	Carbazole	700	U
84-74-2	Di-n-butylphthalate	260	J
206-44-0	Fluoranthene	700	U
129-00-0	Pyrene	700	U
85-68-7	Butylbenzylphthalate	700	U
91-94-1	3,3'-Dichlorobenzidine	700	U
56-55-3	Benzo(a)anthracene	700	U
218-01-9	Chrysene	700	U
117-81-7	Bis(2-Ethylhexyl)phthalate	700	U
117-84-0	Di-n-octylphthalate	700	U
205-99-2	Benzo(b)fluoranthene	700	U
207-08-9	Benzo(k)fluoranthene	700	U
50-32-8	Benzo(a)pyrene	700	U
193-39-5	Indeno(1,2,3-cd)pyrene	700	U
53-70-3	Dibenz(a,h)anthracene	700	U
191-24-2	Benzo(g,h,i)perylene	700	U

Sample Number

BC7KPG

Organics Analysis Data Sheet  
(Page 4)

Tentatively Identified Compounds

CAS Number	Compound Name	Fraction	RT or Scan Number	Estimated Concentration (ug/l or ug/kg)
1 00123-42-2	2-PENTANONE 4-HYDROXY-4-METH	BNA	49	3200 JBA'
2 50-29-3	DDT	↓	1293	310 JN
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1D  
PESTICIDE SOIL ORGANICS ANALYSIS DATA SHEET

EPA SAMPLE NO.

B07KP6

Lab Name: S-CUBED Contract: 32359-51  
 Lab Code: S3 Case No.: 92-321 SAS No.: SDG No.: 3410  
 Matrix: (soil/water) SOIL Lab Sample ID: 3410-01  
 Sample wt/vol: 30 (g/ml) G Lab File ID: E1130-2DB608031  
 %Moisture: 5.67 decanted: (Y/N) N Date Received: 11/05/92  
 Extraction: (SepF/Cont/Sonc) SONC Date Extracted: 11/09/92  
 Concentrated Extract Volume: 10000 (uL) Date Analyzed: 12/01/92  
 Injection Volume: 1.00 (uL) Dilution Factor: 1.00  
 GPC Cleanup: (Y/N) Y pH: 8.95 Sulfur Cleanup: (Y/N) N

CAS NO. COMPOUND CONCENTRATION UNITS:  
 (ug/L or ug/Kg) ug/kg Q

319-84-6	alpha-BHC	1.80	U
319-85-7	beta-BHC	1.80	U
319-86-8	delta-BHC	1.80	U
58-89-9	gamma-BHC (Lindane)	1.80	U
76-44-8	Heptachlor	1.80	U
309-00-2	Aldrin	1.80	U
1024-57-3	Heptachlor epoxide	1.80	U
959-98-8	Endosulfan I	1.80	U
60-57-1	Dieldrin	3.50	U
72-55-9	4,4'-DDE	262	EC
72-20-8	Endrin	3.50	U
33213-65-9	Endosulfan II	3.50	U
72-54-8	4,4'-DDD	3.50	U
1031-07-8	Endosulfan sulfate	3.50	U
50-29-3	4,4'-DDT	341	EC
72-43-5	Methoxychlor	18.0	U
53494-70-5	Endrin ketone	3.50	U
7421-36-3	Endrin Aldehyde	3.50	U
5103-71-9	alpha-Chlordane	1.80	U
5103-74-2	gamma-Chlordane	1.80	U
8001-35-2	Toxaphene	180	U
12674-11-2	Aroclor-1016	35.0	U
11104-28-2	Aroclor-1221	71.0	U
11141-16-5	Aroclor-1232	35.0	U
53469-21-9	Aroclor-1242	35.0	U
12672-29-6	Aroclor-1248	35.0	U
11097-69-1	Aroclor-1254	35.0	U
11096-82-5	Aroclor-1260	35.0	U

1D  
PESTICIDE SOIL ORGANICS ANALYSIS DATA SHEET

EPA SAMPLE NO.

B07KP6DL

Lab Name: S-CUBED Contract: 32359-51  
 Lab Code: S3 Case No.: 92-321 SAS No.: SDG No.: 3410  
 Matrix: (soil/water) SOIL Lab Sample ID: 3410-01DL  
 Sample wt/vol: 30 (g/ml) G Lab File ID: E1130-2DB608065  
 %Moisture: 5.67 decanted: (Y/N) N Date Received: 11/05/92  
 Extraction: (SepF/Cont/Sonc) SONC Date Extracted: 11/09/92  
 Concentrated Extract Volume: 10000 (uL) Date Analyzed: 12/04/92  
 Injection Volume: 1.00 (uL) Dilution Factor: 10.00  
 GPC Cleanup: (Y/N) Y pH: 8.95 Sulfur Cleanup: (Y/N) N

CAS NO. COMPOUND CONCENTRATION UNITS:  
 (ug/L or ug/Kg) ug/kg Q

319-84-6	alpha-BHC	18.0	U
319-85-7	beta-BHC	18.0	U
319-86-8	delta-BHC	18.0	U
58-89-9	gamma-BHC (Lindane)	18.0	U
76-44-8	Heptachlor	18.0	U
309-00-2	Aldrin	18.0	U
1024-57-3	Heptachlor epoxide	18.0	U
959-98-8	Endosulfan I	18.0	U
60-57-1	Dieldrin	35.0	U
72-55-9	4,4'-DDE	264	DC
72-20-8	Endrin	35.0	U
33213-65-9	Endosulfan II	35.0	U
72-54-8	4,4'-DDD	35.0	U
1031-07-8	Endosulfan sulfate	35.0	U
50-29-3	4,4'-DDT	329	DC
72-43-5	Methoxychlor	180	U
53494-70-5	Endrin ketone	35.0	U
7421-36-3	Endrin Aldehyde	35.0	U
5103-71-9	alpha-Chlordane	18.0	U
5103-74-2	gamma-Chlordane	18.0	U
8001-35-2	Toxaphene	1800	U
12674-11-2	Aroclor-1016	350	U
11104-28-2	Aroclor-1221	710	U
11141-16-5	Aroclor-1232	350	U
53469-21-9	Aroclor-1242	350	U
12672-29-6	Aroclor-1248	350	U
11097-69-1	Aroclor-1254	350	U
11096-82-5	Aroclor-1260	350	U

1D  
HERBICIDE ORGANICS ANALYSIS DATA SHEET

EPA SAMPLE NO.

B07KP6

Lab Name: S-CUBED Contract: 32359-51  
 Lab Code: S3 Case No.: 92-321 SAS No.: SDG No.: 3410  
 Matrix: (soil/water) SOIL Lab Sample ID: 3410-01  
 Sample wt/vol: 5 (g/ml) G Lab File ID: H1120-4DB1701077  
 %Moisture: 5.67 decanted: (Y/N) N Date Received: 11/05/92  
 Extraction: (SepF/Cont/Sonc) SEPF Date Extracted: 11/16/92  
 Concentrated Extract Volume: 5000 (uL) Date Analyzed: 11/22/92  
 Injection Volume: 1.00 (uL) Dilution Factor: 1.00  
 GPC Cleanup: (Y/N) N pH: 8.95 Sulfur Cleanup: (Y/N) N

CAS NO. COMPOUND CONCENTRATION UNITS:  
 (ug/L or ug/Kg) ug/kg Q

94-75-7	2,4-D	105	U
94-82-6	2,4-DB	52.6	U
93-76-5	2,4,5-T	26.3	U
93-72-1	2,4,5-TP	26.3	U
88-85-7	Dinoseb	26.3	U
120-36-5	Dichlorprop	105	U
1918-00-9	Dicamba	52.6	U
75-99-0	Dalapon	52.6	U
93-65-2	MCPFP	26300	U
94-74-0	MCPA	26300	U

1D  
PESTICIDE SOIL ORGANICS ANALYSIS DATA SHEET

EPA SAMPLE NO.

B07KP6

Lab Name: S-CUBED Contract: 32359-51  
 Lab Code: S3 Case No.: 92-321 SAS No.: SDG No.: 3410  
 Matrix: (soil/water) SOIL Lab Sample ID: 3410-01  
 Sample wt/vol: 30 (g/ml) G Lab File ID: A1124-6DB1A021  
 %Moisture: 5.67 decanted: (Y/N) N Date Received: 11/05/92  
 Extraction: (SepF/Cont/Sonc) SONC Date Extracted: 11/09/92  
 Concentrated Extract Volume: 10000 (uL) Date Analyzed: 11/25/92  
 Injection Volume: 1.00 (uL) Dilution Factor: 1.00  
 GPC Cleanup: (Y/N) N pH: 8.95 Sulfur Cleanup: (Y/N) N

CAS NO. COMPOUND CONCENTRATION UNITS:  
 (ug/L or ug/Kg) ug/kg Q

115-90-2	Fensulfothion	53.0	U
13194-48-4	Ethoprop	21.2	U
150-50-5	Merphos	21.2	U
2921-88-2	Chlorpyrifos	21.2	U
298-00-0	Parathion-methyl	10.6	U
298-02-2	Phorate	10.6	U
298-04-4	Disulfoton	10.6	U
299-84-3	Ronnel	21.2	U
300-76-5	Naled	21.2	U
327-98-0	Trichloronate	21.2	U
333-41-5	Diazinon	21.2	U
34843-46-4	Tokuthion(Prothiofos)	10.6	U
35400-43-2	Bolstar(Sulprophos)	10.6	U
55-38-9	Fenthion	10.6	U
56-72-4	Coumaphos	53.0	U
62-73-7	Dichlorvos	10.6	U
7786-34-7	Mevinphos	42.4	U
8065-48-3	Dematon-O	42.4	U
8065-48-3A	Dematon-P	42.4	U
86-50-0	Azinphos methyl	53.0	U
961-11-5	Stirophos(Tetrachlorvinphos)	53.0	U

Analyte: TRPH  
 Method: 418.1  
 Technique: IR Spec.  
 DATE: 11/10/92  
 Analyst: CF  
 Instr: P&E IR Spec.  
 Case:  
 Lot(s): 3392,97,99,3409

Smpl Aliquot: 0.020 <sup>(Kg or L)</sup>  
 Final Volume: 0.1 L

Concs: p.p.m.  
 Reagent #1 20  
 #2 40  
 #3 80  
 #4 160  
 #5 300  
 #6

Standards  
 Source: S-CUBED/EL4240  
 Corr. Coef. (0.99990)

Detection Limit 20mg/kg

Std.	Abs	Conc
Blank	0	0
#1	0.03	20
#2	0.062	40
#3	0.126	80
#4	0.245	160
#5	0.46	300
#6		

S-Cubed Sample ID	Client Sample ID	Abs.	Conc. (ug/ml)	Dil. Factor	SAMPLE Conc.	Detection Limit	% Mois.	(mg/kg) Final CONC.
EBS1109A		0	0.0000	1	0.0000	20		0
LCSS1109A		0.205	133.2923	1	666.4616	20		666
EBS1106B		0	0.0000	1	0.0000	20		0
LCSS1106B		0.203	131.9919	1	659.9596	20		660
3397-04RX	S1454070	0	0.0000	1	0.0000	20	5.9	0
3399-04RX	22A10-4	0	0.0000	1	0.0000	20	15.1	0
3392-01RX	S1459180	0.154	100.1318	1	500.6590	20	17.29	605
3392-01REPR	S1459180REP	0.169	109.8849	1	549.4245	20	17.29	664
3409-01	S1454260	0.081	52.6667	1	263.3336	20	14.5	308
3409-02	S1454578	0.109	70.8725	1	354.3625	20	14.9	416
3409-03	S1454261	0	0.0000	1	0.0000	20	8.33	0
3409-04	S1454264	0	0.0000	1	0.0000	20	10.04	0
3409-05	S1454257	0.231	150.1977	5	3754.9424	100	10.3	4186
3409-06	S1454258	0	0.0000	1	0.0000	20	6.94	0
3409-07	S1454449	0.101	65.6709	1	328.3543	20	9.87	364
3409-08	S1454255	0	0.0000	1	0.0000	20	9.7	0
3409-09	S1454076	0	0.0000	1	0.0000	20	13.31	0
3409-10	S1454068	0	0.0000	1	0.0000	20	6.41	0
3410-01	B07KP6	0	0.0000	1	0.0000	20	5.67	0

## U.S. EPA - CLP

1  
INORGANIC ANALYSES DATA SHEET

EPA SAMPLE NO.

3410-01

Lab Name: S\_CUBED\_\_\_\_\_ Contract: 32359-51\_\_\_\_\_

Lab Code: S3\_\_\_\_\_ Case No.: 92231 SAS No.: \_\_\_\_\_ SDG No.: 3410\_\_\_\_\_

Matrix (soil/water): SOIL\_\_\_\_\_ Lab Sample ID: 3410-01\_\_\_\_\_

Level (low/med): LOW\_\_\_\_\_ Date Received: 11/05/92

% Solids: \_\_\_\_\_94.3

Concentration Units (ug/L or mg/kg dry weight): MG/KG

CAS No.	Analyte	Concentration	C	Q	M
7429-90-5	Aluminum	13400	—	—	P
7440-36-0	Antimony	12.7	U	—	P
7440-38-2	Arsenic	10.6	B	—	F
7440-39-3	Barium	157	—	—	P
7440-41-7	Beryllium	0.64	B	—	P
7440-43-9	Cadmium	0.64	B	—	P
7440-70-2	Calcium	16100	—	—	P
7440-47-3	Chromium	21.0	—	—	P
7440-48-4	Cobalt	14.6	—	—	P
7440-50-8	Copper	27.1	—	—	P
7439-89-6	Iron	27800	—	—	P
7439-92-1	Lead	29.9	—	—	F
7439-95-4	Magnesium	7700	—	—	P
7439-96-5	Manganese	571	—	—	P
7439-97-6	Mercury	0.11	U	—	CV
7440-02-0	Nickel	20.8	—	—	P
7440-09-7	Potassium	2330	—	—	P
7782-49-2	Selenium	6.4	U	—	F
7440-22-4	Silver	7.0	—	—	P
7440-23-5	Sodium	539	U	—	P
7440-28-0	Thallium	6.4	U	—	F
7440-62-2	Vanadium	52.2	—	—	P
7440-66-6	Zinc	96.1	—	—	P
_____	_____	_____	—	—	—
_____	_____	_____	—	—	—

Color Before: \_\_\_\_\_ Clarity Before: \_\_\_\_\_ Texture: \_\_\_\_\_

Color After: \_\_\_\_\_ Clarity After: \_\_\_\_\_ Artifacts: \_\_\_\_\_

Comments:

BO7KP6\_\_\_\_\_

FORM I - IN

7/88

002

## ANIONS ANALYSIS

PAGE 1 OF 1

LABORATORY: S-CUBED  
 CLIENT: WHC  
 PROJECT: 92-359  
 LOT #: 3410  
 FILE #: ANI3410S  
 DISK #: ANI1123  
 METHOD NO.: 300.0  
 UNIT: MG/KG

DATA REVIEWER: *AN 12/02/92*  
 PROJECT REVIEWER:  
 CHARGE #: 32359-51  
 DATE SAMPLED: 11-02-92  
 DATE RECEIVED: 11-05-92  
 PREP DATE: 11-09-92  
 DATE ANALYZED: 11-13-92  
 SAMPLE TYPE: SOIL

LAB ID	F	Cl	NO2	Br	NO3	PO4	SO4		
3410-01	1.96	10.9	<del>40.0</del> <i>40.0</i> <del>&lt;0.3</del> <i>&lt;0.3</i>	<del>40.1</del> <i>40.1</i> <del>&lt;0.5</del> <i>&lt;0.5</i>	13.0	1.43	<del>270</del> <i>311</i>		
<del>3410-01D</del> <i>cu</i>		<del>13.2</del> <i>cu</i>					<del>211</del> <i>cu</i>		

SO<sub>4</sub> result was required 10x dilution due to high concentration level. All other anions were reported on straight analysis run. All QC requirement were met. The sample was leached (20gm into 100ml) into DI type II water prior to analysis.







**Golder Associates Inc.**

4104-148th Avenue, NE  
Redmond, WA 98052  
Telephone (206) 883-0777  
Fax (206) 882-5498



June 11, 1993

Our ref: 893-1458  
WHC/O/378

Westinghouse Hanford Company  
Hanford Analytical Services Management  
345 Hills, MSIN H4-29  
Richland, Washington 99352

ATTENTION: Ms. Brianna Colley

RE: NORTH SLOPE ERA DATA VALIDATION, TASK ORDER G-93-58, TRANSMITTAL OF  
DATA VALIDATION PACKAGES

Dear Ms. Colley:

Enclosed is one analytical data package including associated data validation documentation for a North Slope ERA sample analyzed by the S-Cubed laboratory for volatile, semivolatile, chlorinated pesticide/PCB, chlorinated herbicide and phosphate pesticide organic compounds, metals, anions, and total petroleum hydrocarbons.

The data package included in this shipment is 3410-SCU-080. The validation documentation is located at the front of the data package folder.

Please call if you have any questions.

Sincerely,

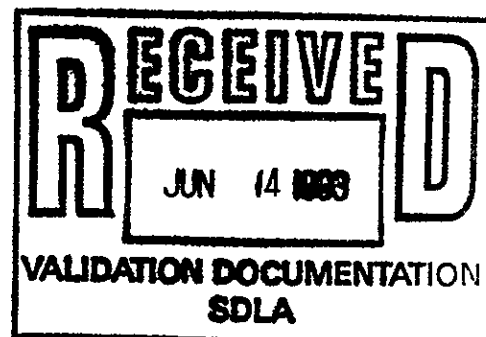
GOLDER ASSOCIATES INC.

  
Kent M. Angelos  
Project Manager

  
Donald M. Caldwell  
Project Director

Enclosures

cc: Bob Henckel, WHC



## MEMORANDUM

TO: North Slope ERA Project QA Record

June 10, 1993

FR: Christina Jensen, Golder Associates Inc.

RE: Volatile Organic Analysis Data Validation Summary for 3410-SCU-080

### INTRODUCTION

This memo presents the results of data validation on data package 3410-SCU-080 consisting of one soil sample submitted for volatile organic analysis. The sample was analyzed by the S-Cubed laboratory using EPA method 8140. The sample identification number, collection date, and sample media are described in the following table.

SAMPLE ID	SAMPLE DATE	MEDIA
B07KP6	11/02/92	SOIL

Data validation was conducted in accordance with the WHC statement of work (WHC 1991) and validation procedures (Bechtold 1992). Attachments 1 through 4 to this memo provide the data validation supporting documentation and a summary of the validated results.

### DATA QUALITY OBJECTIVES

**Precision.** Goals for precision were met with the exception of the evaluation of matrix spike and matrix spike duplicate samples, which the laboratory did not analyze.

**Accuracy.** Goals for accuracy were not evaluated because the laboratory did not analyze a matrix spike and matrix spike duplicate. The samples were not qualified based on this anomaly.

**Sample Result Verification.** All sample results were supported in the raw data with no data correction necessary.

**Detection Limits.** Detection limit goals were met.

**Completeness.** The data package was complete for all requested analyses. A total of one (1) sample was validated in this data set with a total of 33 determinations reported. Out of the 33 determinations reported, all determinations were deemed valid which results in a completeness of 100 percent. This completeness percentage meets the work plan objectives of 90%.

### MAJOR DEFICIENCIES

The were no major deficiencies identified during validation.

## MINOR DEFICIENCIES

### Blanks:

Methylene chloride and acetone were detected in the method blank at 3 ug/kg and 5 ug/kg, respectively. Therefore, the associated sample results which are less than five times the respective blank concentration have been qualified as undetected (U).

## REFERENCES

WHC, 1993, Westinghouse Hanford Company, North Slope ERA Data Validation, Statement of Work, Revision 0, May 1993. Westinghouse Hanford Company, Richland, Washington.

Bechtold, 1992, Westinghouse Hanford Company, Data Validation Procedures for Chemical Analyses, WHC-SD-EN-SPP-002, Rev. 1, 1992. Westinghouse Hanford Company, Richland, Washington.

ATTACHMENT 1  
GLOSSARY OF DATA REPORTING QUALIFIERS

## GLOSSARY OF ORGANIC DATA REPORTING QUALIFIERS

- B -** Indicates the compound was analyzed for and detected in the associated blank. The "B" qualifier for organic data is applied by the laboratory only and is not applied by the data validators.
- U -** Indicates the compound was analyzed for and not detected. The value reported is the sample quantitation limit corrected for sample dilution and moisture content by the laboratory. The data are usable for decision making purposes.
- UJ -** Indicates the compound or analyte was analyzed for and not detected. Due to a quality control deficiency identified during data validation the value reported may not accurately reflect the sample quantitation limit. The data are usable for decision making purposes.
- J -** Indicates the compound or analyte was analyzed for and detected. The associated value is estimated due to a quality control deficiency identified during data validation. The data are usable for decision making purposes.
- UR -** Indicates the compound was analyzed for and not detected; however, due to an identified quality control deficiency the data are unusable.
- R -** Indicates the compound was analyzed for and detected; however, due to an identified quality control deficiency the data are unusable.
- NJ -** Indicates presumptive evidence of a compound at an estimated value.
- N -** Indicates presumptive evidence of a compound.

ATTACHMENT 2  
SUMMARY OF DATA QUALIFICATIONS

**B-7**



ATTACHMENT 3  
AS QUALIFIED DATA SUMMARY

1A  
VOLATILE ORGANICS ANALYSIS DATA SHEET

EPA SAMPLE NO.

B07KP6

Lab Name: S-CUBED Contract: 32359-51  
Lab Code: S3 Case No.: 92-321 SAS No.: SDG No.: 3410  
Matrix: (soil/water) SOIL Lab Sample ID: 3410-01  
Sample wt/vol: 5.00 (g/ml) G Lab File ID: A1110061  
Level: (low/med) LOW Date Received: 11/05/92  
%Moisture: not dec. 5.67 Date Analyzed: 11/10/92  
GC Column: PACK ID: 2.00 (mm) Dilution Factor: 1.00  
Soil Extract Volume: (uL) Soil Aliquot Volume: (uL)

CONCENTRATION UNITS:

CAS NO. COMPOUND (ug/L or ug/Kg) ug/kg

Q

74-87-3	Chloromethane	11	U
74-83-9	Bromomethane	11	U
75-01-4	Vinyl Chloride	11	U
75-00-3	Chloroethane	11	U
75-09-2	Methylene Chloride	11 6	JB
67-64-1	Acetone	11 7	JB
75-15-0	Carbon Disulfide	11	U
75-35-4	1,1-Dichloroethene	11	U
75-34-3	1,1-Dichloroethane	11	U
540-59-0	1,2-Dichloroethene (total)	11	U
67-66-3	Chloroform	11	U
107-06-2	1,2-Dichloroethane	11	U
78-93-3	2-Butanone	11	U
71-55-6	1,1,1-Trichloroethane	11	U
56-23-5	Carbon Tetrachloride	11	U
75-27-4	Bromodichloromethane	11	U
78-87-5	1,2-Dichloropropane	11	U
10061-01-5	cis-1,3-Dichloropropene	11	U
79-01-6	Trichloroethene	11	U
124-48-1	Dibromochloromethane	11	U
79-00-5	1,1,2-Trichloroethane	11	U
71-43-2	Benzene	11	U
10061-02-6	trans-1,3-Dichloropropene	11	U
75-25-2	Bromoform	11	U
108-10-1	4-Methyl-2-pentanone	11	U
591-78-6	2-Hexanone	11	U
127-18-4	Tetrachloroethene	11	U
79-34-5	1,1,2,2-Tetrachloroethane	11	U
108-88-3	Toluene	11	U
108-90-7	Chlorobenzene	11	U
100-41-4	Ethyl Benzene	11	U
100-42-5	Styrene	11	U
1330-20-7	Xylene (total)	11	U

u  
u

Sample Number  
BC7KPG

Organics Analysis Data Sheet  
(Page 4)

Tentatively Identified Compounds

CAS Number	Compound Name	Fraction	RT or Scan Number	Estimated Concentration (ug/l or ug/kg)
1.	NO PIC'S FOUND	VOA		
2.				
3.				
4.				
5.				
6.				
7.				
8.				
9.				
10.				
11.				
12.				
13.				
14.				
15.				
16.				
17.				
18.				
19.				
20.				
21.				
22.				
23.				
24.				
25.				
26.				
27.				
28.				
29.				
30.				

00:

ATTACHMENT 4  
DATA VALIDATION SUPPORTING DOCUMENTATION

## VOLATILE ORGANIC DATA VALIDATION CHECKLIST - FORM A-1

PROJECT: North Slope ERA	REVIEWER: G	DATE: 6/4/98
LABORATORY: S-Cubed	CASE: 92-321	SDG: 3410
SAMPLES/MATRIX: Soil BOTKPL		

## 1. DATA PACKAGE COMPLETENESS

Review the data package for completeness and check off the items below. If any data review elements are missing contact the laboratory for submittal.

<u>Data Package Item</u>	<u>Present?:</u>	<u>Yes</u>	<u>No</u>	<u>N/A</u>
Case Narrative		✓		
Data Summary		✓	✓	
Chain-of-Custody		✓		
QC Summary				
Surrogate report		✓		
MS/MSD report		✓	✓	
Blank summary report		✓		
GC/MS tuning report		✓		
Internal standard summary report		✓		
Sample Data				
Sample reports		✓		
TIC reports for each sample		✓		
RIC reports for all samples		✓		
Raw and corrected spectra for all detected results		✓		
Raw and corrected library search data for all reported TIC		✓		
Quantitation and calculation data for all TIC		✓		
Standards Data				
Initial calibration report		✓		
RIC and quantitation reports for initial calibration		✓		
Continuing calibration reports		✓		
RIC and quantitation reports for cont. calibrations		✓		
Internal standard summary report		✓		
Raw QC Data				
Tuning report, spectra and mass lists		✓		
Blank analysis reports		✓		
TIC reports for all blanks		✓		
RIC and quantitation reports for blanks		✓		
Raw and corrected spectra for all detected results in blanks		✓		
Raw and corrected library search data for all reported TIC				✓

<u>Data Package Item</u>	<u>Present?:</u>	<u>Yes</u>	<u>No</u>	<u>N/A</u>
Quantitation and calculation data for all TIC		<u>—</u>	<u>✓</u>	<u>—</u>
MS/MSD report forms		<u>—</u>	<u>✓</u>	<u>—</u>
RIC and quantitation reports for MS/MSD		<u>—</u>	<u>✓</u>	<u>—</u>
<b>Additional Data</b>				
Moisture/% solids data sheets		<u>—</u>	<u>✓</u>	<u>—</u>
Reduction formulae		<u>—</u>	<u>✓</u>	<u>—</u>
Instrument time logs		<u>—</u>	<u>✓</u>	<u>—</u>
Chemist notebook pages		<u>—</u>	<u>✓</u>	<u>—</u>
Sample preparation sheets		<u>—</u>	<u>✓</u>	<u>—</u>

## 2. HOLDING TIMES

Complete the holding time summary form listing all samples and dates of collection and analysis.

Were all samples analyzed within holding time? Yes No N/A

**ACTION:** If any holding times were exceeded, but not by greater than a factor of two, qualify associated samples as estimated (J for detects or UJ for nondetects), otherwise reject all nondetects (R) and qualify all associated detects as estimated (J).

## 3. INSTRUMENT CALIBRATION, TUNING AND PERFORMANCE CHECKS

### 3.1 GC/MS TUNING AND PERFORMANCE CHECKS

Is a bromofluorobenzene tune report present for each applicable 12-h period? Yes No N/A

Do all tunes on all instruments meet the tuning criteria? Yes No N/A

Do all tunes on all instruments meet the expanded criteria? Yes No N/A

Has the laboratory made any calculation or transcription errors? Yes No N/A

Have the proper significant figures been reported? Yes No N/A

**ACTION:** If the mass calibration is out of specification but within the expanded criteria, qualify associated data as estimated (J for detects or UJ for nondetects). If all tuning criteria are missed, qualify all associated data as unusable (R).

### 3.2 INITIAL CALIBRATION

Is an initial calibration report provided for all instruments? Yes No N/A

Are all RSD values  $\leq 30\%$  (2/88 SOW)? Yes No N/A

Are all RRF values  $\geq 0.05$  (2/88 SOW)? Yes No N/A

Are all applicable RSD values $\leq 20.5\%$ (3/90 SOW)?	<input checked="" type="radio"/> Yes	No	N/A
Are all applicable RSD values $\leq 40\%$ (3/90 SOW)?	Yes	No	<input checked="" type="radio"/> N/A
Are all applicable RRF values within SOW limits (3/90 SOW)?	<input checked="" type="radio"/> Yes	No	N/A
Are all erratic performance compound RRF values $\geq 0.01$ (3/90 SOW)?	<input checked="" type="radio"/> Yes	No	<input checked="" type="radio"/> N/A

ACTION: With the exception of compounds that exhibit erratic performance and making allowances for up to two TCL compounds, if any RRF value is out of specification qualify all detected results for the particular compound as estimated (J) and all nondetects as unusable (R). Making allowances for up to two TCL compounds, if any RSD value is out of specification qualify all associated data as estimated (J for detects or UJ for nondetects).

### 3.3. CONTINUING CALIBRATION

Is a continuing calibration report present for all 12-h periods in which associated samples were analyzed?	<input checked="" type="radio"/> Yes	No	N/A
Are all RRF values $\geq 0.05$ (2/88 SOW)?	Yes	No	<input checked="" type="radio"/> N/A
Are all %D values $\leq 25\%$ (2/88 or 3/90 SOW)?	<input checked="" type="radio"/> Yes	No	N/A
Are all %D values $\leq 40\%$ (3/90 SOW)?	Yes	No	<input checked="" type="radio"/> N/A
Are all RRF values within SOW limits (3/90 SOW)?	<input checked="" type="radio"/> Yes	No	N/A
Are all erratic performance compound RRF values $\geq 0.01$ (3/90 SOW)?	<input checked="" type="radio"/> Yes	No	N/A

ACTION: With the exception of compounds that exhibit erratic performance and making allowances for up to two TCL compounds, if any RRF value is out of specification qualify all associated detected results as estimated and all nondetects as unusable (R). Making allowances for up to two TCL compounds, if any %D is out of specification, qualify all associated results as estimated (J for detects or UJ for nondetects).

## 4. BLANKS

### 4.1 LABORATORY BLANKS

Has the laboratory conducted a method blank analysis per matrix for every 12-h period in which samples were analyzed?	<input checked="" type="radio"/> Yes	No	N/A
Are TCL compounds present in the laboratory blanks?	<input checked="" type="radio"/> Yes	No	N/A

ACTION: Qualify all sample results  $\leq 10$  times the highest blank concentration for the common laboratory contaminants, as nondetects (U) or at the SQL if the result is  $< CRQL$ . Qualify all remaining sample results  $\leq 5$  times the blank concentration in similar fashion.

#### 4.2. FIELD BLANKS

Are TCL compounds present in the field blanks?

Yes No N/A

**ACTION:** Qualify all detected sample results  $\leq 5$  times the amount in any valid field blank as nondetects (U) and note the field blank results in the validation narrative.

#### 5. ACCURACY

##### 5.1 SURROGATE/SYSTEM MONITORING COMPOUND RECOVERY

Are any surrogate recoveries out of specification?

Yes No N/A

Are any surrogate recoveries  $< 10\%$ ?

Yes No N/A

Are any method blank surrogate recoveries out of specification?

Yes No N/A

**ACTION:** Qualify all associated sample results as estimated (J for detects or UJ for nondetects) for surrogates out of specification but  $> 10\%$ . Qualify all associated positive sample results as estimated (J) and all nondetect results as unusable (R) for all surrogates below 10%. If method blank surrogates are out of specification and the associated sample surrogates are acceptable no qualification is necessary, however, the laboratory should be contacted for an explanation.

##### 5.2 MATRIX SPIKE RECOVERY

Has an MS/MSD analysis been conducted per matrix in the sample group?

Yes No N/A

Are MS/MSD recoveries within specification?

Yes No N/A

Are there any calculation errors?

Yes No N/A

**ACTION:** If an MS/MSD analysis has not been conducted contact the laboratory for an explanation. Review the MS/MSD recoveries in conjunction with other QC data such as surrogate recoveries and note the results in the validation narrative. If MS/MSD recoveries are out of specification and sample concentration is  $> 5$  times the spike concentration, no qualification is required, otherwise qualify results as follows: Qualify positive results for the specific class of compound (aromatics and non-aromatics) as estimated (J) in all samples if associated surrogates are also out of specification. The qualification shall only be done on samples of similar matrix as the MS/MSD samples. If it is determined from the review that only the spiked samples are affected by low recoveries, qualify only the results for the spiked sample as described above. If it is determined from the review that out of specification MS/MSD recoveries are indicative of systematic problems in the laboratory such as sample preparation or sample-specific matrix interferences this must be noted in the validation narrative along with the potential affect on the sample results.

*See comment 1*



## 5.3 PERFORMANCE AUDIT SAMPLES

Are the performance audit sample results within the acceptance limits?

Yes

No

N/A

ACTION: Note the results of the performance audit sample in the validation narrative.

## 6. PRECISION

## 6.1 MATRIX SPIKE/MATRIX SPIKE DUPLICATES

Are RPD values within specification?

Yes

No

N/A

Are there any calculation errors?

Yes

No

N/A

ACTION: Review the MS/MSD results in conjunction with other QC data such as field duplicates and note the results in the validation narrative. If MS/MSD RPDs are out of specification and sample results are  $> 5 \times \text{CRQL}$  qualify positive results for the specific class of compound (aromatics and non-aromatics) as estimated (J). If it is determined from the review that out of specification MS/MSD results are indicative of systematic problems in the laboratory such as sample preparation or sample-specific matrix interferences this must be noted in the validation narrative along with the potential affect on the sample results.

## 6.2 FIELD DUPLICATE SAMPLES

Are field duplicate RPD values acceptable?

Yes

No

N/A

ACTION: Note the results of the field duplicate samples in the validation narrative.

## 6.3 FIELD SPLIT SAMPLES

Are field split RPD values acceptable?

Yes

No

N/A

ACTION: Note the results of the field split samples in the validation narrative.

## 7. SYSTEM PERFORMANCE

## 7.1 INTERNAL STANDARDS PERFORMANCE

Are any internal standard area counts outside the acceptance limits?

Yes

No

N/A

Are retention times for any internal standard outside the  $\pm 30$  second windows established by the most recent calibration check?

Yes

No

N/A

ACTION: If the area counts are outside the acceptance limits qualify all associated results as estimated (J for detects or UJ for nondetects). If it is determined from the review that out of specification area counts and relative retention times are indicative of systematic problems within the laboratory the reviewer may consider rejection of all affected sample data (R).

## 8. COMPOUND IDENTIFICATION AND QUANTITATION

## 8.1 COMPOUND IDENTIFICATION

Are detected compounds within  $\pm 0.06$  relative retention time units of the associated calibration standard?

Yes

No

(N/A)

Are all ions at a relative intensity of  $\geq 10\%$  in the standard spectra present in the sample spectra?

Yes

No

(N/A)

Do the relative intensities between the standard and sample spectra agree within 20%?

Yes

No

(N/A)

Have all ions  $> 10\%$  in the sample spectra that are not present in the standard spectra been reviewed for possible background contamination?

Yes

No

(N/A)

Are molecular ions present in the reference spectrum present in the sample spectrum?

Yes

No

(N/A)

ACTION: If compound identification is in error and retention time and mass spectral criteria are exceeded qualify all affected positive results as unusable (R). If cross-contamination between analyses is suspected, qualify affected data as unusable (R). Note the results in the validation narrative.

## 8.2 REPORTED RESULTS AND QUANTITATION LIMITS

Has the laboratory used the correct RRF values and internal standard(s) for quantitation?

(Yes)

No

N/A

Are results and quantitation limits calculated properly?

(Yes)

No

N/A

Has the laboratory reported the sample quantitation limits within  $5 \times \text{CRQL}$  values?

(Yes)

No

N/A

ACTION: If the results and quantitation limits are in error contact the laboratory for clarification and note in the validation narrative.

## 8.3 TENTATIVELY IDENTIFIED COMPOUNDS (TIC)

Has the laboratory conducted a spectral library search on all candidate TIC peaks in accordance with the analytical SOW?

(Yes)

No

N/A

Has the laboratory properly identified and coded all TIC?

Yes

No

(N/A)

ACTION: If the laboratory has failed to search the minimum number of TIC peaks in the chromatogram contact the laboratory for submittal of the required data. Qualify as nondetects (U) all TIC compounds present in samples and blanks using the review criteria specified in the validation requirements. If TIC identification is in error sample results should be qualified as nondetects (U) or unusable (R). If TIC identifications are judged valid, qualify the results as presumptive and estimated (JN).

9. OVERALL ASSESSMENT AND SUMMARY

Has the laboratory conducted the analysis in accordance with the analytical SOW?

☒ Yes    No    N/A

Were project specific data quality objectives met for this analysis?

☒ Yes    No    N/A

**ACTION:** Summarize all the data qualifications recommended in the foregoing sections, and complete the data validation narrative according to the requirements of Section 10.0 of the data validation requirements.

COMMENTS (attach additional sheets as necessary):

1. A matrix spike / matrix spike duplicate was not performed for this SDG. No qualification of the data will be made because of this.

2. The detected compounds have been qualified as N because of blank contamination.

## HOLDING TIME SUMMARY - FORM B-1

[illegible]

## BLANK AND SAMPLE DATA SUMMARY - FORM B-3

[illegible]

## MEMORANDUM

TO: North Slope ERA Project QA Record

June 10, 1993

FR: Christina Jensen, Golder Associates Inc.

RE: Total Recoverable Petroleum Hydrocarbon Analysis Data Validation Summary for 3410-SCU-080

### INTRODUCTION

This memo presents the results of data validation on data package 3410-SCU-080 consisting of one soil sample submitted for total recoverable petroleum hydrocarbon (TRPH) analysis. The sample was analyzed by the S-Cubed laboratory using EPA method 418.1. The sample identification number, collection date, and sample media are described in the following table.

SAMPLE ID	SAMPLE DATE	MEDIA
B07KP6	11/02/92	SOIL

Data validation was conducted in accordance with the WHC statement of work (WHC 1991) and validation procedures (Bechtold 1992). Attachments 1 through 4 to this memo provide the data validation supporting documentation and a summary of the validated results.

### DATA QUALITY OBJECTIVES

**Precision.** Goals for precision were met.

**Accuracy.** Goals for accuracy were met.

**Sample Result Verification.** All sample results were supported in the raw data with no data correction necessary.

**Detection Limits.** Detection limit goals were met.

**Completeness.** The data package was complete for all requested analyses. A total of one sample was validated in this data set with a total of one determination reported. Out of the one determination reported, it was deemed valid which results in a completeness of 100 percent. This completeness percentage meets the work plan objectives of 90%.

### MAJOR DEFICIENCIES

There were no major deficiencies identified requiring rejection of the data.

## MINOR DEFICIENCIES

There were no minor deficiencies identified requiring rejection of the data.

## REFERENCES

WHC, 1993, Westinghouse Hanford Company, North Slope ERA Data Validation, Statement of Work, Revision 0, May 1993. Westinghouse Hanford Company, Richland, Washington.

Bechtold, 1992, Westinghouse Hanford Company, Data Validation Procedures for Chemical Analyses, WHC-SD-EN-SPP-002, Rev. 1, 1992. Westinghouse Hanford Company, Richland, Washington.



ATTACHMENT 1  
GLOSSARY OF DATA REPORTING QUALIFIERS

## GLOSSARY OF ORGANIC DATA REPORTING QUALIFIERS

- B - Indicates the compound was analyzed for and detected in the associated blank. The "B" qualifier for organic data is applied by the laboratory only and is not applied by the data validators.
- U - Indicates the compound was analyzed for and not detected. The value reported is the sample quantitation limit corrected for sample dilution and moisture content by the laboratory. The data are usable for decision making purposes.
- UJ - Indicates the compound or analyte was analyzed for and not detected. Due to a quality control deficiency identified during data validation the value reported may not accurately reflect the sample quantitation limit. The data are usable for decision making purposes.
- J - Indicates the compound or analyte was analyzed for and detected. The associated value is estimated due to a quality control deficiency identified during data validation. The data are usable for decision making purposes.
- UR - Indicates the compound was analyzed for and not detected; however, due to an identified quality control deficiency the data are unusable.
- R - Indicates the compound was analyzed for and detected; however, due to an identified quality control deficiency the data are unusable.
- NJ - Indicates presumptive evidence of a compound at an estimated value.
- N - Indicates presumptive evidence of a compound.

ATTACHMENT 2  
SUMMARY OF DATA QUALIFICATIONS

**B-7**

ATTACHMENT 3  
AS QUALIFIED DATA SUMMARY

Analyte: TRPH  
 Method: 418.1  
 Technique: IR Spec.  
 DATE: 11/10/92  
 Analyst: CF  
 Instr: P&E IR Spec.  
 Case:  
 Lot(s): 3392,97,99,3409

Smpl Aliquot:  
 Final Volume:

0.020 Kg or L  
 0.1 L

Concs: p.p.m.  
 Reagent #1 20  
 #2 40  
 #3 80  
 #4 160  
 #5 300  
 #6

Standards  
 Source: S-CUBED/EL4240  
 Corr. Coef. (0.99990)

Detection Limit 20mg/kg

Std.	Abs	Conc
Blank	0	0
#1	0.03	20
#2	0.062	40
#3	0.126	80
#4	0.245	160
#5	0.46	300
#6		

S-Cubed Sample ID	Client Sample ID	Abs.	Conc. (ug/ml)	Dil. Factor	SAMPLE Conc.	Detection Limit	% Mois.	(mg/kg) Final CONC.
EBS1109A		0	0.0000	1	0.0000	20		0
LCSS1109A		0.205	133.2923	1	666.4616	20		666
EBS1106B		0	0.0000	1	0.0000	20		0
LCSS1106B		0.203	131.9919	1	659.9596	20		660
3397-04RX	S1454070	0	0.0000	1	0.0000	20	5.9	0
3399-04RX	22A10-4	0	0.0000	1	0.0000	20	15.1	0
3392-01RX	S1459180	0.154	100.1318	1	500.6590	20	17.29	605
3392-01REPR	S1459180REP	0.169	109.8849	1	549.4245	20	17.29	664
3409-01	S1454260	0.081	52.6667	1	263.3336	20	14.5	308
3409-02	S1454578	0.109	70.8725	1	354.3625	20	14.9	416
3409-03	S1454261	0	0.0000	1	0.0000	20	8.33	0
3409-04	S1454264	0	0.0000	1	0.0000	20	10.04	0
3409-05	S1454257	0.231	150.1977	5	3754.9424	100	10.3	4186
3409-06	S1454258	0	0.0000	1	0.0000	20	6.94	0
3409-07	S1454449	0.101	65.6709	1	328.3543	20	9.87	364
3409-08	S1454255	0	0.0000	1	0.0000	20	9.7	0
3409-09	S1454076	0	0.0000	1	0.0000	20	13.31	0
3409-10	S1454068	0	0.0000	1	0.0000	20	6.41	0
3410-01	B07KP6	0	0.0000	1	0.0000	20	5.67	0

50/4/43

ATTACHMENT 4

DATA VALIDATION SUPPORTING DOCUMENTATION

TRPH 26/9/93

## VOLATILE ORGANIC DATA VALIDATION CHECKLIST - FORM A-1

PROJECT: 11/11/11 Stone ERA	REVIEWER: 5	DATE: 10/9/93
LABORATORY: S-Cubed	CASE: 92-321	SDG: 3410
SAMPLES/MATRIX: 5001 B27KDL		

## 1. DATA PACKAGE COMPLETENESS

Review the data package for completeness and check off the items below. If any data review elements are missing contact the laboratory for submittal.

Data Package Item	Present?:	Yes	No	N/A
Case Narrative		<input checked="" type="checkbox"/>		
Data Summary		<input type="checkbox"/>	<input checked="" type="checkbox"/>	
Chain-of-Custody		<input checked="" type="checkbox"/>		
QC Summary				
Surrogate report	9/10/93	<input checked="" type="checkbox"/>		<input checked="" type="checkbox"/>
MS/MSD report		<input checked="" type="checkbox"/>		<input checked="" type="checkbox"/>
Blank summary report		<input checked="" type="checkbox"/>		<input checked="" type="checkbox"/>
GC/MS tuning report		<input type="checkbox"/>		<input checked="" type="checkbox"/>
Internal standard summary report		<input type="checkbox"/>		<input checked="" type="checkbox"/>
Sample Data				
Sample reports		<input checked="" type="checkbox"/>		
TIC reports for each sample		<input type="checkbox"/>		<input checked="" type="checkbox"/>
RIC reports for all samples		<input type="checkbox"/>		<input checked="" type="checkbox"/>
Raw and corrected spectra for all detected results		<input type="checkbox"/>		<input checked="" type="checkbox"/>
Raw and corrected library search data for all reported TIC		<input type="checkbox"/>		<input checked="" type="checkbox"/>
Quantitation and calculation data for all TIC		<input type="checkbox"/>		<input checked="" type="checkbox"/>
Standards Data				
Initial calibration report		<input checked="" type="checkbox"/>		
RIC and quantitation reports for initial calibration		<input type="checkbox"/>		<input checked="" type="checkbox"/>
Continuing calibration reports		<input type="checkbox"/>		<input checked="" type="checkbox"/>
RIC and quantitation reports for cont. calibrations		<input type="checkbox"/>		<input checked="" type="checkbox"/>
Internal standard summary report		<input type="checkbox"/>		<input checked="" type="checkbox"/>
Raw QC Data				
Tuning report, spectra and mass lists		<input checked="" type="checkbox"/>		<input checked="" type="checkbox"/>
Blank analysis reports		<input checked="" type="checkbox"/>		<input checked="" type="checkbox"/>
TIC reports for all blanks		<input type="checkbox"/>		<input checked="" type="checkbox"/>
RIC and quantitation reports for blanks		<input type="checkbox"/>		<input checked="" type="checkbox"/>
Raw and corrected spectra for all detected results in blanks		<input type="checkbox"/>		<input checked="" type="checkbox"/>
Raw and corrected library search data for all reported TIC		<input type="checkbox"/>		<input checked="" type="checkbox"/>



<u>Data Package Item</u>	<u>Present?:</u>	<u>Yes</u>	<u>No</u>	<u>N/A</u>
Quantitation and calculation data for all TIC				<input checked="" type="checkbox"/>
MS/MSD report forms	SEE COMMENT	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>
RIC and quantitation reports for MS/MSD		<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>
<b>Additional Data</b>				
Moisture/% solids data sheets		<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>
Reduction formulae		<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>
Instrument time logs		<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>
Chemist notebook pages		<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>
Sample preparation sheets		<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>

## 2. HOLDING TIMES

Complete the holding time summary form listing all samples and dates of collection and analysis.

Were all samples analyzed within holding time? ☒ Yes ☐ No ☐ N/A

ACTION: If any holding times were exceeded, but not by greater than a factor of two, qualify associated samples as estimated (J for detects or UJ for nondetects), otherwise reject all nondetects (R) and qualify all associated detects as estimated (J).

## 3. INSTRUMENT CALIBRATION, TUNING AND PERFORMANCE CHECKS

### 3.1 GC/MS TUNING AND PERFORMANCE CHECKS

Is a bromofluorobenzene tune report present for each applicable 12-h period? Yes No ☒ N/A

Do all tunes on all instruments meet the tuning criteria? Yes No ☒ N/A

Do all tunes on all instruments meet the expanded criteria? Yes No ☒ N/A

Has the laboratory made any calculation or transcription errors? Yes ☒ No ☒ N/A

Have the proper significant figures been reported? Yes No ☒ N/A

ACTION: If the mass calibration is out of specification but within the expanded criteria, qualify associated data as estimated (J for detects or UJ for nondetects). If all tuning criteria are missed, qualify all associated data as unusable (R).

### 3.2 INITIAL CALIBRATION

Is an initial calibration report provided for all instruments? ☒ Yes ☐ No ☐ N/A

Are all RSD values  $\leq 30\%$  (2/88 SOW)? Yes No ☒ N/A

Are all RRF values  $\geq 0.05$  (2/88 SOW)? Yes No ☒ N/A

Are all applicable RSD values $\leq 20.5\%$ (3/90 SOW)?	Yes	No	(N/A)
Are all applicable RSD values $\leq 40\%$ (3/90 SOW)?	Yes	No	(N/A)
Are all applicable RRF values within SOW limits (3/90 SOW)?	Yes	No	(N/A)
Are all erratic performance compound RRF values $\geq 0.01$ (3/90 SOW)?	Yes	No	(N/A)

**ACTION:** With the exception of compounds that exhibit erratic performance and making allowances for up to two TCL compounds, if any RRF value is out of specification qualify all detected results for the particular compound as estimated (J) and all nondetects as unusable (R). Making allowances for up to two TCL compounds, if any RSD value is out of specification qualify all associated data as estimated (J for detects or UJ for nondetects).

### 3.3. CONTINUING CALIBRATION

Is a continuing calibration report present for all 12-h periods in which associated samples were analyzed?	Yes	No	(N/A)
Are all RRF values $\geq 0.05$ (2/88 SOW)?	Yes	No	(N/A)
Are all %D values $\leq 25\%$ (2/88 or 3/90 SOW)?	Yes	No	(N/A)
Are all %D values $\leq 40\%$ (3/90 SOW)?	Yes	No	(N/A)
Are all RRF values within SOW limits (3/90 SOW)?	Yes	No	(N/A)
Are all erratic performance compound RRF values $\geq 0.01$ (3/90 SOW)?	Yes	No	(N/A)

**ACTION:** With the exception of compounds that exhibit erratic performance and making allowances for up to two TCL compounds, if any RRF value is out of specification qualify all associated detected results as estimated and all nondetects as unusable (R). Making allowances for up to two TCL compounds, if any %D is out of specification, qualify all associated results as estimated (J for detects or UJ for nondetects).

## 4. BLANKS

### 4.1 LABORATORY BLANKS

Has the laboratory conducted a method blank analysis per matrix for every 12-h period in which samples were analyzed?	(Yes)	No	N/A
Are TCL compounds present in the laboratory blanks?	Yes	(No)	N/A

**ACTION:** Qualify all sample results  $\leq 10$  time the highest blank concentration for the common laboratory contaminants, as nondetects (U) or at the SQL if the result is  $< CRQL$ . Qualify all remaining sample results  $\leq 5$  times the blank concentration in similar fashion.

## 4.2. FIELD BLANKS

Are TCL compounds present in the field blanks? Yes No N/A

ACTION: Qualify all detected sample results  $\leq 5$  times the amount in any valid field blank as nondetects (U) and note the field blank results in the validation narrative.

## 5. ACCURACY

## 5.1 SURROGATE/SYSTEM MONITORING COMPOUND RECOVERY

Are any surrogate recoveries out of specification? Yes No N/A

Are any surrogate recoveries  $< 10\%$ ? Yes No N/A

Are any method blank surrogate recoveries out of specification? Yes No N/A

ACTION: Qualify all associated sample results as estimated (J for detects or UJ for nondetects) for surrogates out of specification but  $> 10\%$ . Qualify all associated positive sample results as estimated (J) and all nondetect results as unusable (R) for all surrogates below  $10\%$ . If method blank surrogates are out of specification and the associated sample surrogates are acceptable no qualification is necessary, however, the laboratory should be contacted for an explanation.

## 5.2 MATRIX SPIKE RECOVERY

<sup>LCS</sup>  
Has an MS/MSD analysis been conducted per matrix in the sample group? Yes No N/A

<sup>LCS</sup>  
Are MS/MSD recoveries within specification? Yes No N/A

Are there any calculation errors? Yes No N/A

ACTION: If an MS/MSD analysis has not been conducted contact the laboratory for an explanation. Review the MS/MSD recoveries in conjunction with other QC data such as surrogate recoveries and note the results in the validation narrative. If MS/MSD recoveries are out of specification and sample concentration is  $> 5$  times the spike concentration, no qualification is required, otherwise qualify results as follows: Qualify positive results for the specific class of compound (aromatics and non-aromatics) as estimated (J) in all samples if associated surrogates are also out of specification. The qualification shall only be done on samples of similar matrix as the MS/MSD samples. If it is determined from the review that only the spiked samples are affected by low recoveries, qualify only the results for the spiked sample as described above. If it is determined from the review that out of specification MS/MSD recoveries are indicative of systematic problems in the laboratory such as sample preparation or sample-specific matrix interferences this must be noted in the validation narrative along with the potential affect on the sample results.

## 5.3 PERFORMANCE AUDIT SAMPLES

Are the performance audit sample results within the acceptance limits?

Yes No N/A

ACTION: Note the results of the performance audit sample in the validation narrative.

## 6. PRECISION

## 6.1 MATRIX SPIKE/MATRIX SPIKE DUPLICATES

Are RPD values within specification?

Yes No N/A

Are there any calculation errors?

Yes No N/A

ACTION: Review the MS/MSD results in conjunction with other QC data such as field duplicates and note the results in the validation narrative. If MS/MSD RPDs are out of specification and sample results are  $> 5 \times \text{CRQL}$  qualify positive results for the specific class of compound (aromatics and non-aromatics) as estimated (J). If it is determined from the review that out of specification MS/MSD results are indicative of systematic problems in the laboratory such as sample preparation or sample-specific matrix interferences this must be noted in the validation narrative along with the potential affect on the sample results.

## 6.2 FIELD DUPLICATE SAMPLES

Are field duplicate RPD values acceptable?

Yes No N/A

ACTION: Note the results of the field duplicate samples in the validation narrative.

## 6.3 FIELD SPLIT SAMPLES

Are field split RPD values acceptable?

Yes No N/A

ACTION: Note the results of the field split samples in the validation narrative.

## 7. SYSTEM PERFORMANCE

## 7.1 INTERNAL STANDARDS PERFORMANCE

Are any internal standard area counts outside the acceptance limits?

Yes No N/A

Are retention times for any internal standard outside the  $\pm 30$  second windows established by the most recent calibration check?

Yes No N/A

ACTION: If the area counts are outside the acceptance limits qualify all associated results as estimated (J for detects or UJ for nondetects). If it is determined from the review that out of specification area counts and relative retention times are indicative of systematic problems within the laboratory the reviewer may consider rejection of all affected sample data (R).

## 8. COMPOUND IDENTIFICATION AND QUANTITATION

## 8.1 COMPOUND IDENTIFICATION

Are detected compounds within  $\pm 0.06$  relative retention time units of the associated calibration standard?

Yes No N/A

Are all ions at a relative intensity of  $\geq 10\%$  in the standard spectra present in the sample spectra?

Yes No N/A

Do the relative intensities between the standard and sample spectra agree within 20%?

Yes No N/A

Have all ions  $> 10\%$  in the sample spectra that are not present in the standard spectra been reviewed for possible background contamination?

Yes No N/A

Are molecular ions present in the reference spectrum present in the sample spectrum?

Yes No N/A

ACTION: If compound identification is in error and retention time and mass spectral criteria are exceeded qualify all affected positive results as unusable (R). If cross-contamination between analyses is suspected, qualify affected data as unusable (R). Note the results in the validation narrative.

## 8.2 REPORTED RESULTS AND QUANTITATION LIMITS

Has the laboratory used the correct RRF values and internal standard(s) for quantitation?

Yes No N/A

Are results and quantitation limits calculated properly?

Yes No N/A

Has the laboratory reported the sample quantitation limits within  $5 \times \text{CRQL}$  values?

Yes No N/A

ACTION: If the results and quantitation limits are in error contact the laboratory for clarification and note in the validation narrative.

## 8.3 TENTATIVELY IDENTIFIED COMPOUNDS (TIC)

Has the laboratory conducted a spectral library search on all candidate TIC peaks in accordance with the analytical SOW?

Yes No N/A

Has the laboratory properly identified and coded all TIC?

Yes No N/A

ACTION: If the laboratory has failed to search the minimum number of TIC peaks in the chromatogram contact the laboratory for submittal of the required data. Qualify as nondetects (U) all TIC compounds present in samples and blanks using the review criteria specified in the validation requirements. If TIC identification is in error sample results should be qualified as nondetects (U) or unusable (R). If TIC identifications are judged valid, qualify the results as presumptive and estimated (JN).

9. OVERALL ASSESSMENT AND SUMMARY

Has the laboratory conducted the analysis in accordance with the analytical SOW?

☒ Yes    No    N/A

Were project specific data quality objectives met for this analysis?

☒ Yes    No    N/A

**ACTION:** Summarize all the data qualifications recommended in the foregoing sections, and complete the data validation narrative according to the requirements of Section 10.0 of the data validation requirements.

COMMENTS (attach additional sheets as necessary):

1. The laboratory analyzed an LCS vialled on  
in MS/MSD, which will be reviewed for  
% recovery.

## HOLDING TIME SUMMARY - FORM B-1

[illegible]



## MEMORANDUM

TO: North Slope ERA Project QA Record

June 10, 1993

FR: Christina Jensen, Golder Associates Inc.

RE: Semivolatile Organics Analysis Data Validation Summary for 3410-SCU-080

### INTRODUCTION

This memo presents the results of data validation on data package 3410-SCU-080 consisting of one soil sample submitted for semivolatile organics analysis. The sample was analyzed by the S-Cubed laboratory using CLP protocols. The sample identification, collection date, and sample media are described in the following table.

SAMPLE ID	SAMPLE DATE	MEDIA
B07KP6	11/02/92	SOIL

Data validation was conducted in accordance with the WHC statement of work (WHC 1991) and validation procedures (Bechtold 1992). Attachments 1 through 4 to this memo provide the data validation supporting documentation and a summary of the validated results.

### DATA QUALITY OBJECTIVES

**Precision.** Goals for precision were met with the exception of the evaluation of matrix spike (MS) and matrix spike duplicate (MSD) samples, which were not analyzed by the laboratory.

**Accuracy.** Goals for accuracy were met with the exception of the evaluation of MS and MSD samples, which were not analyzed by the laboratory.

**Sample Result Verification.** All sample results were supported in the raw data with no data correction necessary. The laboratory performed an unknown search and identified DDT in sample B07KP4 at a concentration of 310 ug/kg and confirmed this in the pesticides/PCB analysis at a concentration of 341 ug/kg.

**Detection Limits.** Detection limit goals were met.

**Completeness.** The data package was complete for all requested analyses. A total of one (1) sample was validated in this data set with a total of 64 determinations reported. Out of the 64 determinations reported, all determinations were deemed valid which results in a completeness of 100 percent. This completeness percentage meets the work plan objectives of 90%.

### MAJOR DEFICIENCIES

A tentatively identified compound (TIC), an aldol condensation product, 4-hydroxy-4-methyl-2-pentanone was detected in sample B07KP6 at a concentration of 3200 ug/kg and was qualified as unusable (R).

#### MINOR DEFICIENCIES

The laboratory indicated in the case narrative that a matrix spike (MS) and matrix spike duplicate (MSD) were analyzed for this sample set, however, the data and forms were not submitted with the data package. Therefore, the data was not evaluated based on precision and accuracy using the MS/MSD results.

#### REFERENCES

WHC, 1993, Westinghouse Hanford Company, North Slope ERA Data Validation, Statement of Work, Revision 0, May 1993. Westinghouse Hanford Company, Richland, Washington.

Bechtold, 1992, Westinghouse Hanford Company, Data Validation Procedures for Chemical Analyses, WHC-SD-EN-SPP-002, Rev. 1, 1992. Westinghouse Hanford Company, Richland, Washington.

ATTACHMENT 1

GLOSSARY OF DATA REPORTING QUALIFIERS

## GLOSSARY OF ORGANIC DATA REPORTING QUALIFIERS

- B - Indicates the compound was analyzed for and detected in the associated blank. The "B" qualifier for organic data is applied by the laboratory only and is not applied by the data validators.
- U - Indicates the compound was analyzed for and not detected. The value reported is the sample quantitation limit corrected for sample dilution and moisture content by the laboratory. The data are usable for decision making purposes.
- UJ - Indicates the compound or analyte was analyzed for and not detected. Due to a quality control deficiency identified during data validation the value reported may not accurately reflect the sample quantitation limit. The data are usable for decision making purposes.
- J - Indicates the compound or analyte was analyzed for and detected. The associated value is estimated due to a quality control deficiency identified during data validation. The data are usable for decision making purposes.
- UR - Indicates the compound was analyzed for and not detected; however, due to an identified quality control deficiency the data are unusable.
- R - Indicates the compound was analyzed for and detected; however, due to an identified quality control deficiency the data are unusable.
- NJ - Indicates presumptive evidence of a compound at an estimated value.
- N - Indicates presumptive evidence of a compound.

ATTACHMENT 2  
SUMMARY OF DATA QUALIFICATIONS



ATTACHMENT 3  
AS QUALIFIED DATA SUMMARY

1B  
SEMIVOLATILE ORGANICS ANALYSIS DATA SHEET

EPA SAMPLE NO.

B07KP6

Lab Name: S-CUBED Contract: 32359-51  
Lab Code: S3 Case No.: 92-321 SAS No.: SDG No.: 3410  
Matrix: (soil/water) SOIL Lab Sample ID: 3410-01  
Sample wt/vol: 30 (g/ml) G Lab File ID: DN12061  
Level: (low/med) LOW Date Received: 11/05/92  
%Moisture: 5.67 decanted: (Y/N) N Date Extracted: 11/09/92  
Concentrated Extract Volume: 2000.00 (uL) Date Analyzed: 11/12/92  
Injection Volume: 1.00 (uL) Dilution Factor: 1.00  
GPC Cleanup: (Y/N) Y pH: 8.95

CAS NO. COMPOUND CONCENTRATION UNITS:  
(ug/L or ug/Kg) ug/kg Q

108-95-2	Phenol	700	U
111-44-4	bis(2-Chloroethyl) ether	700	U
95-57-8	2-Chlorophenol	700	U
541-73-1	1,3-Dichlorobenzene	700	U
106-46-7	1,4-Dichlorobenzene	700	U
95-50-1	1,2-Dichlorobenzene	700	U
95-48-7	2-Methylphenol	700	U
108-60-1	2,2'-oxybis(1-Chloropropane)	700	U
106-44-5	4-Methylphenol	700	U
621-64-7	N-Nitroso-di-n-propylamine	700	U
67-72-1	Hexachloroethane	700	U
98-95-3	Nitrobenzene	700	U
78-59-1	Isophorone	700	U
88-75-5	2-Nitrophenol	700	U
105-67-9	2,4-Dimethylphenol	700	U
111-91-1	bis(2-Chloroethoxy) methane	700	U
120-83-2	2,4-Dichlorophenol	700	U
120-82-1	1,2,4-Trichlorobenzene	700	U
91-20-3	Naphthalene	700	U
106-47-8	4-Chloroaniline	700	U
87-68-3	Hexachlorobutadiene	700	U
59-50-7	4-Chloro-3-methylphenol	700	U
91-57-6	2-Methylnaphthalene	700	U
77-47-4	Hexachlorocyclopentadiene	700	U
88-06-2	2,4,6-Trichlorophenol	700	U
95-95-4	2,4,5-Trichlorophenol	1700	U
91-58-7	2-Chloronaphthalene	700	U
88-74-4	2-Nitroaniline	1700	U
131-11-3	Dimethylphthalate	700	U
208-96-8	Acenaphthylene	700	U
606-20-2	2,6-Dinitrotoluene	700	U
99-09-2	3-Nitroaniline	1700	U
83-32-9	Acenaphthene	700	U



1C  
SEMIVOLATILE ORGANICS ANALYSIS DATA SHEET

EPA SAMPLE NO.

B07KP6

Lab Name: S-CUBED Contract: 32359-51  
Lab Code: S3 Case No.: 92-321 SAS No.: SDG No.: 3410  
Matrix: (soil/water) SOIL Lab Sample ID: 3410-01  
Sample wt/vol: 30 (g/ml) G Lab File ID: DN12061  
Level: (low/med) LOW Date Received: 11/05/92  
%Moisture: 5.67 decanted: (Y/N) N Date Extracted: 11/09/92  
Concentrated Extract Volume: 2000.00 (uL) Date Analyzed: 11/12/92  
Injection Volume: 1.00 (uL) Dilution Factor: 1.00  
GPC Cleanup: (Y/N) Y pH: 8.95

CAS NO. COMPOUND CONCENTRATION UNITS:  
(ug/L or ug/Kg) ug/kg Q

51-28-5	2,4-Dinitrophenol	1700	U
100-02-7	4-Nitrophenol	1700	U
132-64-9	Dibenzofuran	700	U
121-14-2	2,4-Dinitrotoluene	700	U
84-66-2	Diethylphthalate	700	U
7005-72-3	4-Chlorophenyl-phenyl ether	700	U
86-73-7	Fluorene	700	U
100-01-6	4-Nitroaniline	1700	U
534-52-1	4,6-Dinitro-2-methylphenol	1700	U
86-30-6	N-Nitrosodiphenylamine (1)	700	U
101-55-3	4-Bromophenyl-phenylether	700	U
118-74-1	Hexachlorobenzene	700	U
87-86-5	Pentachlorophenol	1700	U
85-01-8	Phenanthrene	700	U
120-12-7	Anthracene	700	U
86-74-8	Carbazole	700	U
84-74-2	Di-n-butylphthalate	260	J
206-44-0	Fluoranthene	700	U
129-00-0	Pyrene	700	U
85-68-7	Butylbenzylphthalate	700	U
91-94-1	3,3'-Dichlorobenzidine	700	U
56-55-3	Benzo(a)anthracene	700	U
218-01-9	Chrysene	700	U
117-81-7	Bis(2-Ethylhexyl)phthalate	700	U
117-84-0	Di-n-octylphthalate	700	U
205-99-2	Benzo(b)fluoranthene	700	U
207-08-9	Benzo(k)fluoranthene	700	U
50-32-8	Benzo(a)pyrene	700	U
193-39-5	Indeno(1,2,3-cd)pyrene	700	U
53-70-3	Dibenz(a,h)anthracene	700	U
191-24-2	Benzo(g,h,i)perylene	700	U

Sample Number

EC7KPG

Organics Analysis Data Sheet  
(Page 4)

Tentatively Identified Compounds

CAS Number	Compound Name	Fraction	RT or Scan Number	Estimated Concentration (ug/l or ug/kg)
1. 00123-42-2	2-PENTANOIC 4-HYDROXY-4-METH	BNA	49	3200 FAT
2. 50-29-3	DDT	↓	1293	310 JN
3.				
4.				
5.				
6.				
7.				
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ATTACHMENT 4

DATA VALIDATION SUPPORTING DOCUMENTATION

## SEMI-VOLATILE ORGANIC DATA VALIDATION CHECKLIST - FORM A-2

PROJECT: <i>North Slope ERA</i>	REVIEWER: <i>CJ</i>	DATE: <i>6/7/93</i>
LABORATORY: <i>S-Cubed</i>	CASE: <i>92-321</i>	SDG: <i>3410</i>
SAMPLES/MATRIX: <i>soil B07KPG</i>		

## 1. DATA PACKAGE COMPLETENESS

Review the data package for completeness and check off the items below. If any data review elements are missing contact the laboratory for submittal.

<u>Data Package Item</u>	Present?:	Yes	No	N/A
Case Narrative		<input checked="" type="checkbox"/>		
Data Summary		<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/> <i>Final Report</i>	
Chain-of-Custody		<input checked="" type="checkbox"/>		
QC Summary		<input checked="" type="checkbox"/>		
Surrogate report		<input checked="" type="checkbox"/>		
MS/MSD report		<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/> <i>See comment:</i>	
Blank summary report		<input checked="" type="checkbox"/>		
GC/MS tuning report		<input checked="" type="checkbox"/>		
Internal standard summary report		<input checked="" type="checkbox"/>		
Sample Data				
Sample reports		<input checked="" type="checkbox"/>		
TIC reports for each sample		<input checked="" type="checkbox"/>		
RIC reports for all samples		<input checked="" type="checkbox"/>		
Raw and corrected spectra for all detected results		<input checked="" type="checkbox"/>		
Raw and corrected library search data for all reported TIC		<input checked="" type="checkbox"/>		
Quantitation and calculation data for all TIC		<input checked="" type="checkbox"/>		
Standards Data				
Initial calibration report		<input checked="" type="checkbox"/>		
RIC and quantitation reports for initial calibration		<input checked="" type="checkbox"/>		
Continuing calibration reports		<input checked="" type="checkbox"/>		
RIC and quantitation reports for cont. calibrations		<input checked="" type="checkbox"/>		
Internal standard summary report		<input checked="" type="checkbox"/>		
Raw QC Data				
Tuning report, spectra and mass lists		<input checked="" type="checkbox"/>		
Blank analysis reports		<input checked="" type="checkbox"/>		
TIC reports for all blanks		<input checked="" type="checkbox"/>		
RIC and quantitation reports for blanks		<input checked="" type="checkbox"/>		
Raw and corrected spectra for all detected results in blanks		<input checked="" type="checkbox"/>		
Raw and corrected library search data for all reported TIC		<input checked="" type="checkbox"/>		
Quantitation and calculation data for all TIC		<input checked="" type="checkbox"/>		
MS/MSD report forms		<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	

<u>Data Package Item</u>	<u>Present?:</u>	<u>Yes</u>	<u>No</u>	<u>N/A</u>
RIC and quantitation reports for MS/MSD	—	—	✓	—
Additional Data				
Moisture/% solids data sheets	—	—	✓	—
Reduction formulae	—	—	✓	—
Instrument time logs	—	—	✓	—
Chemist notebook pages	—	—	✓	—
Sample preparation sheets	✓	—	—	—

## 2. HOLDING TIMES

Were all samples extracted within holding time?	(Yes)	No	N/A
Were all samples analyzed within holding time?	(Yes)	No	N/A

ACTION: If any holding times were exceeded, but not by greater than a factor of two, qualify associated samples as estimated (J for detects or UJ for nondetects), otherwise reject all nondetects (R) and qualify all associated detects as estimated (J).

## 3. INSTRUMENT CALIBRATION, TUNING AND PERFORMANCE CHECKS

### 3.1 GC/MS TUNING AND PERFORMANCE CHECKS

Is a DFTPP tune report present for each applicable 12h period?	(Yes)	No	N/A
Do all tunes on all instruments meet the tuning criteria?	(Yes)	No	N/A
Do all tunes on all instruments meet the expanded criteria?	Yes	No	(N/A)
Has the laboratory made any calculation or transcription errors?	Yes	(No)	N/A
Have the proper significant figures been reported?	(Yes)	No	N/A

ACTION: If the mass calibration is out of specification but within the expanded criteria, qualify associated data as estimated (J for detects and UJ for nondetects). If all tuning criteria are not met, qualify all associated data as unusable (R).

### 3.2 INITIAL CALIBRATION

Is an initial calibration report provided for all instruments?	(Yes)	No	N/A
Are all RSD values $\leq 30\%$ (2/88 SOW)?	Yes	No	(N/A)
Are all RRF values $\geq 0.05$ (2/88 SOW)?	Yes	No	(N/A)
Are all applicable RSD values $\leq 20.5\%$ (3/90 SOW)?	(Yes)	No	N/A
Are all applicable RSD values $\leq 40\%$ (3/90 SOW)?	Yes	No	(N/A)

Are all applicable RRF values within SOW limits (3/90 SOW)? ☒ Yes No N/A

Are all erratic performance compound RRF values  $\geq 0.01$  (3/90 SOW)? ☒ Yes No N/A

**ACTION:** With the exception of compounds that exhibit erratic performance and making allowances for up to four TCL compounds or surrogates, if any RRF value is out of specification qualify all detected results for the particular compound as estimated (J) and all nondetects as unusable (R). Making allowances for up to four TCL compounds or surrogates, if any RSD value is out of specification qualify all associated data as estimated (J for detects or UJ for nondetects).

### 3.3. CONTINUING CALIBRATION

Is a continuing calibration report present for all 12-h periods in which associated samples were analyzed? ☒ Yes No N/A

Are all RRF values  $\geq 0.05$  (2/88 SOW)? Yes No ☒ N/A

Are all %D values  $\leq 25\%$  (2/88 or 3/90 SOW)? ☒ Yes No N/A

Are all %D values  $\leq 40\%$  (3/90 SOW)? Yes No ☒ N/A

Are all RRF values within SOW limits (3/90 SOW)? ☒ Yes No N/A

Are all erratic performance compound RRF values  $\geq 0.01$  (3/90 SOW)? ☒ Yes No N/A

**ACTION:** With the exception of compounds that exhibit erratic performance and making allowances for up to four TCL compounds or surrogates, if any RRF value is out of specification qualify all associated detected results as estimated and all nondetects as unusable (R). Making allowances for up to four TCL compounds or surrogates, if any %D is out of specification, qualify all associated results as estimated (J for detects or UJ for nondetects).

## 4. BLANKS

### 4.1 LABORATORY BLANKS

Has the laboratory conducted a method blank analysis per matrix for every extraction batch? ☒ Yes No N/A

Are compounds reported in the laboratory blanks? Yes ☒ No N/A

**ACTION:** Qualify all sample results  $< 10$  times the highest blank concentration for the common laboratory contaminants, as nondetects (U) or at the SQL if the result is  $< \text{CRQL}$ . Qualify all remaining sample results  $< 5$  times the blank concentration in similar fashion.

## 4.2. FIELD BLANKS

Are compounds reported in the field blanks?

Yes No N/A

ACTION: Qualify all detected sample results  $\leq 5$  times the amount in any valid field blank as nondetects (U) and note the results of the field blanks in the validation narrative.

## 5. ACCURACY

## 5.1 SURROGATE RECOVERY/SYSTEM MONITORING COMPOUND RECOVERY

Are any surrogate recoveries out of specification?

Yes No N/AAre any surrogate recoveries  $< 10\%$ ?Yes No N/A

Are any method blank surrogate recoveries out of specification?

Yes No N/A

ACTION: Qualify all associated data as estimated (J for detects and UJ for nondetects) if at least two semivolatile surrogates are out of specification. If any surrogate is below 10% recovery qualify associated detected results as estimated (J) and associated nondetect results as unusable (R). If method blank surrogates are out of specification and associated sample surrogates are acceptable no qualification is required, however, the laboratory should be contacted for an explanation.

## 5.2 MATRIX SPIKE RECOVERY

Has an MS/MSD analysis been conducted per matrix in the sample group?

*See comment 1*  
Yes No N/A

Are MS/MSD recoveries within specification?

Yes No N/A

Are there any calculation errors?

Yes No N/A

ACTION: If an MS/MSD analysis has not been conducted contact the laboratory for an explanation. Review the MS/MSD recoveries in conjunction with other QC data such as surrogate recoveries and note the results in the validation narrative. If MS/MSD recoveries are out of specification and sample concentration is  $> 5$  times the spike concentration, no qualification is required, otherwise qualify results as follows: Qualify positive results for the specific class of compound (aromatics and non-aromatics) as estimated (J) in all samples if associated surrogates are also out of specification. The qualification shall only be done on samples of similar matrix as the MS/MSD samples. If it is determined from the review that only the spiked samples are affected by low recoveries, qualify only the results for the spiked sample as described above. If it is determined from the review that out of specification MS/MSD recoveries are indicative of systematic problems in the laboratory such as sample preparation or sample-specific matrix interferences this must be noted in the validation narrative along with the potential affect on the sample results.

## 5.3 PERFORMANCE AUDIT SAMPLES

Are the results for the performance audit samples within the acceptance limits?

Yes

No

(N/A)

ACTION: Note the results of the performance audit samples in the validation narrative.

## 6. PRECISION

## 6.1 MATRIX SPIKE/MATRIX SPIKE DUPLICATES

Are all RPD values within specification?

Yes

No

(N/A)

Are there any calculation errors?

Yes

No

(N/A)

ACTION: Review the MS/MSD results in conjunction with other QC data such as field duplicates and note the results in the validation narrative. If MS/MSD RPDs are out of specification and sample results are  $> 5 \times \text{CRQL}$  qualify positive results for the specific class of compound (aromatics and non-aromatics) as estimated (J). If it is determined from the review that out of specification MS/MSD results are indicative of systematic problems in the laboratory such as sample preparation or sample-specific matrix interferences this must be noted in the validation narrative along with the potential affect on the sample results.

## 6.2 FIELD DUPLICATE SAMPLES

Are field duplicate RPD values acceptable?

Yes

No

(N/A)

ACTION: Note the results of the field duplicate samples in the validation narrative.

## 6.3 FIELD SPLIT SAMPLES

Are field split RPD values acceptable?

Yes

No

(N/A)

ACTION: Note the results of the field split samples in the validation narrative.

## 7. SYSTEM PERFORMANCE

## 7.1 INTERNAL STANDARDS PERFORMANCE

Are any internal standard area counts outside the acceptance limits?

Yes

(No)

N/A

Are retention times for any internal standard outside the  $\pm 30$  second windows established by the most recent calibration check?

Yes

(No)

N/A

ACTION: If the area counts are outside the acceptance limits qualify all associated results as estimated (J for detects and UJ for nondetects. If it is determined from the review that out of specification area counts and relative retention times are indicative of systematic problems within the laboratory the reviewer may consider rejection of all affected sample data (R).



## 8. COMPOUND IDENTIFICATION AND QUANTITATION

## 8.1 COMPOUND IDENTIFICATION

Are detected compounds within  $\pm 0.06$  relative retention time units of the associated calibration standard?

Yes

No

N/A

*See comment 2*

Are all ions at a relative intensity of  $\geq 10\%$  in the standard spectra present in the sample spectra?

Yes

No

N/A

Do the relative intensities between the standard and sample spectra agree within 20%?

Yes

No

N/A

Have all ions  $> 10\%$  in the sample spectra that are not present in the standard spectra been reviewed for possible background contamination?

Yes

No

N/A

Are molecular ions in the reference spectrum present in the sample spectrum?

Yes

No

N/A

ACTION: If compound identification is in error and retention time and mass spectral criteria are exceeded qualify all affected positive results as unusable (R). If cross-contamination between analyses is suspected, qualify affected data as unusable (R).

## 8.2 REPORTED RESULTS AND QUANTITATION LIMITS

Has the laboratory used the correct RRF values and internal standards for quantitation?

Yes

No

N/A

Are results and quantitation limits calculated properly?

Yes

No

N/A

Has the laboratory reported the sample quantitation limits within 5xCRQL values?

Yes

No

N/A

ACTION: If the quantitation limits are in error contact the laboratory for clarification and note in the validation narrative.

## 8.3 TENTATIVELY IDENTIFIED COMPOUNDS

Has the laboratory conducted a spectral library search on all candidate TIC peaks in accordance with the analytical SOW?

Yes

No

N/A

Has the laboratory properly identified and coded all TIC?

Yes

No

N/A

*See comment 3.*

ACTION: If the laboratory has failed to search the minimum number of TIC peaks in the chromatogram contact the laboratory for submittal of the required data. Qualify as nondetects (U) all TIC compounds present in samples and blanks using the review criteria specified in the validation requirements. If TIC identification is in error sample results should be qualified as nondetects (U) or unusable (R). If TIC identifications are judged valid, qualify the results as presumptive and estimated (JN).

9. OVERALL ASSESSMENT AND SUMMARY

Has the laboratory conducted the analysis in accordance with the analytical SOW?

☒ Yes    No    N/A

Were project specific data quality objectives met for this analysis?

☒ Yes    No    N/A

**ACTION:** Summarize all the data qualifications and complete the data validation narrative as specified in Section 10.0 of the data validation requirements.

## COMMENTS (attach additional sheets as necessary):

1. The case narrative says that a matrix spiking solution problem is present, however, the laboratory did not submit MS/MSD results or forms with this data package. No data qualification will be done as a result of the missing MS/MSD data.

2. Di-n-butyl phthalate was detected in ~~the~~ sample bottle but the retention times were not listed out. The spectra was reviewed and identification of the compound is correct.

3. DDT was identified in the TIC search. The compound was also detected in the Pest/PCB analyses.

TIC concentration = 310 ug/kg Pest/PCB concentration = 341 ug/kg

4-hydroxy-4-methyl-2-pentanone was detected in the sample at 3200 ug/kg, which is an aldol condensation product. The result is qualified as unusable (R)

## HOLDING TIME SUMMARY - FORM B-1

[illegible]

## MEMORANDUM

TO: North Slope ERA Project QA Record

June 11, 1993

FR: Christina Jensen, Golder Associates Inc.

RE: Organochlorine Pesticide/PCB Data Validation Summary for 3410-SCU-080

### INTRODUCTION

This memo presents the results of data validation on data package 3410-SCU-080 consisting of one soil sample submitted for organochlorine pesticide/PCB analysis. The sample was analyzed by the S-Cubed laboratory using CLP protocols. The sample identification number, collection date, and sample media are described in the following table.

SAMPLE ID	SAMPLE DATE	MEDIA
B07KP6	11/02/92	SOIL

Data validation was conducted in accordance with the WHC statement of work (WHC 1991) and validation procedures (Bechtold 1992). Attachments 1 through 4 to this memo provide the data validation supporting documentation and a summary of the validated results.

### DATA QUALITY OBJECTIVES

**Precision.** Goals for precision could not be evaluated because a matrix spike (MS) and matrix spike duplicate (MSD) were not analyzed.

**Accuracy.** A MS and MSD were not analyzed for this sample delivery group (SDG). A laboratory control sample was analyzed and reviewed for percent recovery with all results acceptable.

The surrogate percent recoveries were outside control limits as noted in "Minor Deficiencies".

**Sample Result Verification.** The retention time for DDD shifted during sample analysis, therefore the laboratory did not report the result. The DDD was confirmed by GCMS and was corrected on the result form for sample B07KP6 and is designated by a "C" qualifier.

The laboratory reported the original concentrations instead of the diluted concentrations for 4,4-DDE and 4,4-DDT which exceeded the calibration range. The results form for sample B07KP6 was corrected to reflect the diluted sample concentrations. The difference between the original and diluted concentrations is small.

**Detection Limits.** The laboratory performed a cleanup (GPC) on the sample and the reported detection limits did not reflect this. Therefore, the detection limits were multiplied by a factor of two and the result form was corrected.

**Completeness.** The data package was complete for all requested analyses. A total of one sample was validated in this data set with a total of 28 determinations reported. Out of the 28 determinations reported, all determinations were deemed valid which results in a completeness of 100 percent. This completeness percentage meets the work plan objectives of 90%.

## MAJOR DEFICIENCIES

There were no major deficiencies identified requiring rejection of the data.

## MINOR DEFICIENCIES

### Surrogates

All surrogate recoveries for method blank EBS1109 were slightly low (51 to 58%). Since the recoveries for sample B07KP6 were acceptable, no qualification or results was made.

## REFERENCES

WHC, 1993, Westinghouse Hanford Company, North Slope ERA Data Validation, Statement of Work, Revision 0, May 1993. Westinghouse Hanford Company, Richland, Washington.

Bechtold, 1992, Westinghouse Hanford Company, Data Validation Procedures for Chemical Analyses, WHC-SD-EN-SPP-002, Rev. 1, 1992. Westinghouse Hanford Company, Richland, Washington.

ATTACHMENT 1

GLOSSARY OF DATA REPORTING QUALIFIERS

## GLOSSARY OF ORGANIC DATA REPORTING QUALIFIERS

- B - Indicates the compound was analyzed for and detected in the associated blank. The "B" qualifier for organic data is applied by the laboratory only and is not applied by the data validators.
- U - Indicates the compound was analyzed for and not detected. The value reported is the sample quantitation limit corrected for sample dilution and moisture content by the laboratory. The data are usable for decision making purposes.
- UJ - Indicates the compound or analyte was analyzed for and not detected. Due to a quality control deficiency identified during data validation the value reported may not accurately reflect the sample quantitation limit. The data are usable for decision making purposes.
- J - Indicates the compound or analyte was analyzed for and detected. The associated value is estimated due to a quality control deficiency identified during data validation. The data are usable for decision making purposes.
- UR - Indicates the compound was analyzed for and not detected; however, due to an identified quality control deficiency the data are unusable.
- R - Indicates the compound was analyzed for and detected; however, due to an identified quality control deficiency the data are unusable.
- NJ - Indicates presumptive evidence of a compound at an estimated value.
- N - Indicates presumptive evidence of a compound.



ATTACHMENT 2  
SUMMARY OF DATA QUALIFICATIONS

## DATA QUALIFICATION SUMMARY - FORM B-7

[illegible]

ATTACHMENT 3  
AS QUALIFIED DATA SUMMARY

1D  
PESTICIDE SOIL ORGANICS ANALYSIS DATA SHEET

EPA SAMPLE NO.

B07KP6

Lab Name: S-CUBED Contract: 32359-51  
Lab Code: S3 Case No.: 92-321 SAS No.: SDG No.: 3410  
Matrix: (soil/water) SOIL Lab Sample ID: 3410-01  
Sample wt/vol: 30 (g/ml) G Lab File ID: E1130-2DB608031  
%Moisture: 5.67 decanted: (Y/N) N Date Received: 11/05/92  
Extraction: (SepF/Cont/Sonc) SONC Date Extracted: 11/09/92  
Concentrated Extract Volume: 10000 (uL) Date Analyzed: 12/01/92  
Injection Volume: 1.00 (uL) Dilution Factor: 1.00  
GPC Cleanup: (Y/N) Y pH: 8.95 Sulfur Cleanup: (Y/N) N

CAS NO. COMPOUND CONCENTRATION UNITS:  
(ug/L or ug/Kg) ug/kg Q

319-84-6	alpha-BHC	3.4 <del>1.80</del>	U
319-85-7	beta-BHC	3.6 <del>1.80</del>	U
319-86-8	delta-BHC	3.4 <del>1.80</del>	U
58-89-9	gamma-BHC (Lindane)	3.6 <del>1.80</del>	U
76-44-8	Heptachlor	3.6 <del>1.80</del>	U
309-00-2	Aldrin	3.6 <del>1.80</del>	U
1024-57-3	Heptachlor epoxide	3.4 <del>1.80</del>	U
959-98-8	Endosulfan I	3.6 <del>1.80</del>	U
60-57-1	Dieldrin	7.0 3.50	U
72-55-9	4,4'-DDE	272 <del>262</del>	EC
72-20-8	Endrin	7.0 <del>3.50</del>	U
33213-65-9	Endosulfan II	7.0 <del>3.50</del>	U
72-54-8	4,4'-DDD	127 <del>3.50</del>	U
1031-07-8	Endosulfan sulfate	7.0 <del>3.50</del>	U
50-29-3	4,4'-DDT	342 <del>341</del>	EC
72-43-5	Methoxychlor	36.0 <del>18.0</del>	U
53494-70-5	Endrin ketone	7.0 <del>3.50</del>	U
7421-36-3	Endrin Aldehyde	7.0 <del>3.50</del>	U
5103-71-9	alpha-Chlordane	3.6 <del>1.80</del>	U
5103-74-2	gamma-Chlordane	3.6 <del>1.80</del>	U
8001-35-2	Toxaphene	3.00 <del>180</del>	U
12674-11-2	Aroclor-1016	70.0 <del>35.0</del>	U
11104-28-2	Aroclor-1221	142 <del>71.0</del>	U
11141-16-5	Aroclor-1232	70.0 <del>35.0</del>	U
53469-21-9	Aroclor-1242	70.0 <del>35.0</del>	U
12672-29-6	Aroclor-1248	70.0 <del>35.0</del>	U
11097-69-1	Aroclor-1254	70.0 <del>35.0</del>	U
11096-82-5	Aroclor-1260	70.0 <del>35.0</del>	U

ATTACHMENT 4

DATA VALIDATION SUPPORTING DOCUMENTATION

## PESTICIDE/PCB DATA VALIDATION CHECKLIST - FORM A-3

PROJECT: 71 North Slope FRA	REVIEWER: C. J. J. J.	DATE: 6/19/98
LABORATORY: S-Cubed	CASE: 92-321	SDG: 3410
SAMPLES/MATRIX: Soil B07KPL6		

## 1. DATA PACKAGE COMPLETENESS

Review the data package for completeness and check off the items below. If any data review elements are missing contact the laboratory for resubmittal.

<u>Data Package Item</u>	<u>Present?:</u>	<u>Yes</u>	<u>No</u>	<u>N/A</u>
Case Narrative		<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Data Summary		<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Chain-of-Custody		<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
QC Summary		<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Surrogate report		<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
MS/MSD report		<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>
Blank summary report		<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Sample Data		<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Sample reports		<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Chromatograms		<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
GC integration reports		<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Worksheets		<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>
UV traces from GPC		<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
GC/MS confirmation spectra		<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Standards Data		<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Pesticides Evaluation Standards Summary		<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Pesticides/PCB Standards Summary		<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Pesticides/PCB identification		<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Pesticides standard chromatograms		<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Raw QC Data		<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Blank analysis report forms and chromatograms		<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
MS/MSD report forms and chromatograms		<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>

<u>Data Package Item</u>	<u>Present?:</u>	<u>Yes</u>	<u>No</u>	<u>N/A</u>
<b>Additional Data</b>				
Moisture/% solids data sheets		—	✓	—
Reduction formulae		—	✓	—
Instrument time logs		—	✓	—
Chemist notebook pages		—	✓	—
Sample preparation sheets		✓	—	—

## 2. HOLDING TIMES

Were all samples extracted within holding time? Yes No N/A

Were all samples analyzed within holding time? Yes No N/A

**ACTION:** If any holding times were exceeded, but not by greater than a factor of two, qualify associated samples as estimated (J for detects or UJ for nondetects), otherwise reject all nondetects (R) and qualify all associated detects as estimated (J).

## 3. INSTRUMENT PERFORMANCE AND CALIBRATIONS

### 3.1 INSTRUMENT PERFORMANCE (2/88 SOW)

Are DDT retention times greater than 12 minutes? Yes No N/A

**ACTION:** If DDT retention time is  $\leq 12$  minutes and resolution is  $< 25\%$  qualify associated data as unusable (R).

Is resolution between DDT peaks acceptable? Yes No N/A

**ACTION:** If resolution between DDT peaks is unacceptable qualify associated data as unusable (R).

Do all pesticide standards elute within the established retention time windows? Yes No N/A

**ACTION:** If the standards do not meet the retention time criteria and peaks are not present near or within the retention time windows no sample qualification is necessary. If peaks are near or within the retention time windows and the standards and matrix spikes do not fall within the expanded retention time windows calculated according to the validation requirements, qualify all associated sample results from the last in-control point as unusable (R).

Are DDT breakdowns  $\leq 20\%$ ? Yes No N/A

**ACTION:** If the DDT percent breakdown exceeds 20%, qualify all detected results for DDT as estimated (J) and all nondetects as unusable (R) if DDD and DDE are detected. In addition qualify all results for DDD or DDE as presumptive and estimated (NJ).

Are endrin breakdowns  $\leq 20\%$ ? Yes No N/A

**ACTION:** If the endrin breakdown exceeds 20%, qualify all detected results for endrin as estimated (J) and all nondetects as unusable (R) if endrin aldehyde or endrin ketone are detected. In addition, qualify all results for endrin ketone as presumptive and estimated (NJ).

Are DBC retention time differences within specification? Yes No (N/A)

**ACTION:** If DBC %D values are outside the limits and the shift is occurring repeatedly in samples and standards, qualify affected sample results as unusable (R).

### 3.2 CALIBRATIONS (2/88 SOW)

Are RSD values for aldrin, endrin, DDT and DBC  $\leq 10\%$ ? Yes No (N/A)

Have all standards been analyzed within 72 h of any sample? Yes No (N/A)

Has a 3-point calibration been conducted for DDT or toxaphene? Yes No (N/A)

Have all standards been analyzed at the start of each 72-h sequence? Yes No (N/A)

Have evaluation standards A, B, and C been analyzed within 72 h of any sample? Yes No (N/A)

Has the confirmation standard mix been analyzed after every five samples? Yes No (N/A)

Has evaluation standard B analyzed every 10 samples? Yes No (N/A)

Are %D values for initial and subsequent standards  $\leq 15\%$  for quantitation standards and  $\leq 20\%$  for confirmation standards? Yes No (N/A)

**ACTION:** If the RSD criteria were exceeded or three point calibrations not conducted qualify associated detects as estimated (J). If all standards were not analyzed at the beginning of each 72-h sequence qualify associated data as unusable (R). If the confirmation standards were not analyzed properly qualify associated detects as estimated (J). If the continuing calibration criteria were not met qualify associated quantitation data as estimated (J).



## 3.3 INSTRUMENT PERFORMANCE AND INITIAL CALIBRATION (3/90 SOW)

Is peak resolution acceptable?

(Yes) No N/A

ACTION: If the resolution criteria are not met, reject positive sample results generated after initial calibration (R).

Are DDT and endrin breakdowns  $\leq 20.0\%$ 

(Yes) No N/A

ACTION: If the breakdown criteria are not met qualify sample results as described in Section 5.3.1 of the validation requirements.

Are single component target compounds in the PEMs, INDA, INDB and the calibration standards within the retention time windows?

(Yes) No N/A

ACTION: If the retention time criteria are not met and no peaks are present in the samples within two times the retention time windows ( $\pm 0.04$ ,  $\pm 0.05$  for methoxychlor), no qualification is necessary. If peaks are present in samples within the retention time window a review is made of the raw data to determine expanded retention time windows (see Section 5.3.1 of the validation requirements). If all standards and matrix spikes fall within the expanded windows then no qualification of sample results is necessary. If all standards and matrix spikes do not fall within the expanded windows then all affected sample results are qualified as unusable (R).

Are the RPDs acceptable for the PEMs?

(Yes) No N/A

ACTION: If the RPD criteria are not met qualify associated positive sample results as estimated (J).

Are the RSDs for the calibration factors  $< 20\%$  ( $< 10.0\%$  for the BHC series, DDT, endrin, and methoxychlor)?

(Yes) No N/A

ACTION: If the RSD criteria are not met qualify associated positive sample results as estimated (J).

## 3.4 CALIBRATION VERIFICATION (3/90 SOW)

Have the analytical sequence requirements been met for the analysis of instrument blanks, PEMs, INDA and INDB mixes?

(Yes) No N/A

ACTION: If the analytical sequence requirements are not followed and any of the resolution or retention time criteria listed below are exceeded, reject associated positive results (R).

Is peak resolution acceptable for PEMs, INDA and INDB mixes?

(Yes) No N/A

ACTION: If the resolution criteria are not met reject positive sample results generated after a noncompliant standard analysis (R).

Are single component target compounds in the PEMs, INDA and INDB mixes within the retention time windows?

(Yes) No N/A

**ACTION:** If the retention time criteria are not met and no peaks are present in the samples analyzed after the noncompliant standard within two times the retention time windows ( $\pm 0.04$ ,  $\pm 0.05$  for methoxychlor), no qualification is necessary. If peaks are present in samples within the expanded windows rejected associated positive and nondetect results (R).

Are RPDs between the calculated and true amounts in the PEMs, INDA and INDB mixes  $\leq 25.0\%$ ?

☒ Yes    No    N/A

**ACTION:** If the RPD criteria are not met qualify associated positive sample results as estimated (J).

Are DDT and endrin breakdowns in the PEMs  $\leq 20.0\%$  ( $\leq 30.0\%$  total combined)?

☒ Yes    No    N/A

**ACTION:** If the breakdown criteria are not met qualify associated positive sample results in accordance with the criteria specified in Section 5.3.1.

#### 4. BLANKS

##### 4.1 LABORATORY BLANKS

Has the laboratory analyzed the method blanks at the required frequency?

☒ Yes    No    N/A

Has the laboratory analyzed a sulfur clean-up blank if required?

Yes    No    ☒ N/A

Has the laboratory analyzed instrument blanks at the required frequency?

Yes    No    ☒ N/A

Are target compounds present in the blanks?

Yes    ☒ No    N/A

**ACTION:** Qualify all associated positive results as nondetects (U) that are  $< 5$  times the highest concentration in any acceptable blank.

##### 4.2 FIELD BLANKS

Are target compounds present in the field blanks?

Yes    No    ☒ N/A

**ACTION:** If target compounds are present in the field blanks qualify all positive sample results  $< 5$  times the highest valid field blank concentrations as nondetects (U) and note the results in the validation narrative.

## 5. ACCURACY

## 5.1 SURROGATE RECOVERY

Are any surrogate recoveries out of specification?

☒ Yes    No    N/A

Do any samples show nondetects for surrogates?

☐ Yes    ☒ No    N/A

Are any method blank surrogates out of specification?

☒ Yes    ☐ No    N/A *comment 2*

**ACTION:** Qualify all associated sample results as estimated (J for detects and UJ for nondetects) for surrogates out of specification. If the surrogate was not detected (0% recovery) in the sample qualify associated nondetects as unusable (R). If method blank surrogates are out of specification and sample surrogates are acceptable, no qualification is required however, the laboratory should be contacted for an explanation.

## 5.2 MATRIX SPIKE RECOVERY

Has the laboratory analyzed a MS/MSD per matrix for the sample group?

☐ Yes    ☒ No    N/A *comment 1*
*LCS*  
Are ~~MS/MSD~~ recoveries within specification?

☒ Yes    No    N/A

Are there any calculation or transcription errors?

☐ Yes    ☒ No    N/A

**ACTION:** If MS/MSD analyses have not been conducted contact the laboratory for clarification. Review the MS/MSD recoveries in conjunction with other QC data such as surrogate recoveries and note the results in the validation narrative. If MS/MSD recoveries are out of specification and sample concentration is > 5 times the spike concentration, no qualification is required, otherwise qualify results as follows: Qualify positive results as estimated (J) in all samples if associated surrogates are also out of specification. The qualification shall only be done on samples of similar matrix as the MS/MSD samples. If it is determined from the review that only the spiked samples are affected by the low recoveries, qualify only the results for the spiked sample as described above. If it is determined from the review that out of specification MS/MSD recoveries are indicative of systematic problems in the laboratory such as sample preparation or sample-specific matrix interferences this must be noted in the validation narrative along with the potential affect on the sample results.

## 5.3 PERFORMANCE AUDIT SAMPLES

Are performance audit sample results within the acceptance limits?

☐ Yes    No    ☒ N/A

**ACTION:** Note the results of the performance audit samples in the validation narrative.

## 6. PRECISION

## 6.1 MATRIX SPIKE/MATRIX SPIKE DUPLICATE SAMPLES

Are the RPD values within specification?

Yes No N/A

ACTION: Review the MS/MSD results in conjunction with other QC data such as field duplicates and note the results in the validation narrative. If MS/MSD RPD values are out of specification and sample results are  $> 5 \times \text{CRQL}$  qualify positive results as estimated (J). If it is determined from the review that out of specification MS/MSD results are indicative of systematic problems in the laboratory such as sample preparation or sample-specific matrix interferences this must be noted in the validation narrative along with the potential affect on the sample results.

## 6.2 FIELD DUPLICATE SAMPLES

Are field duplicate RPD values acceptable?

Yes No N/A

ACTION: Note the results of the field duplicate samples in the validation narrative.

## 6.3 FIELD SPLIT SAMPLES

Are field split RPD values acceptable?

Yes No N/A

ACTION: Note the results of the field split samples in the validation narrative.

## 7. COMPOUND IDENTIFICATION AND QUANTTATION

## 7.1 COMPOUND IDENTIFICATION

Do positive results meet the retention time window criteria?

SCR comment 3  
Yes No N/A

Were positive results analyzed on dissimilar columns?

Yes No N/A

If dieldrin and DDE were reported was a 3% OV-1 column used for confirmation (2/88 SOW data only)?

Yes No N/A

Do retention times and relative peak height ratios match the expected patterns for multipeak compounds (PCB, toxaphene or chlordane)?

Yes No N/AHas GC/MS confirmation been conducted on sample extract concentrations  $> 10$  ppm?Yes No N/A

**ACTION:** If positive results do not meet the retention time criteria qualify all detected results as nondetects as follows: If the misidentified peak is outside the retention time windows and no interferences are noted report the CRQL and if the misidentified peak interferes with a target peak then the report value is qualified as estimated and nondetected (UJ). If positive results were not confirmed on dissimilar columns, reject affected results (R). If a 3% OV-1 was used to confirm dieldrin and DDE, reject the affected data (R). If PCB, chlordane or toxaphene identification is questionable qualify the results as presumptive and estimated (NJ). If GC/MS confirmation was not conducted contact the laboratory for explanation and note in the validation narrative.

## 7.2 REPORTED RESULTS AND QUANTITATION LIMITS

Are results and quantitation limits calculated properly?

(Yes) No N/A

Has the laboratory reported the sample quantitation limits within 5xCRQL values?

See comment 4, 5  
(Yes) No N/A

**ACTION:** If results and quantitation limits are in error contact the laboratory for clarification and note in the validation narrative.

## 8. OVERALL ASSESSMENT AND SUMMARY

Has the laboratory conducted the analysis in accordance with the analytical SOW?

(Yes) No N/A

Were project specific data quality objectives met for this analysis?

(Yes) No N/A

**ACTION:** Summarize all the data qualifications and complete the data validation narrative as specified in Section 10.0 of the data validation requirements.

## COMMENTS (attach additional sheets as necessary):

1. TMS/MSD was not submitted however an LCS was analyzed with the sample batch and will be reviewed for recovery.
2. The surrogate recoveries for the blank (EBS1109) are all out, but not extremely low - the limits are 60-15 and the recoveries are 51 to 58%. No sample qualification will be made because of this.
3. The retention time for DDD shifted however it was detected both for the GC and confirmed by GC/MS. It was not reported on the results form and will be added. The retention time of DDD is outside the windows.
4. The laboratory did a GPC but did not reflect this in the detection limits reported on the results form. The undetected values will be multiplied by 2.
5. The laboratory reported the results that had not been diluted. The <sup>results</sup> form has been changed ~~and~~ to reflect the diluted values. The difference between these two values is small.

## HOLDING TIME SUMMARY - FORM B-1

[illegible]

## MEMORANDUM

TO: North Slope ERA Project QA Record

June 11, 1993

FR: Christina Jensen, Golder Associates Inc.

RE: Organochlorine Herbicide Analysis Data Validation Summary for 3410-SCU-080

### INTRODUCTION

This memo presents the results of data validation on data package 3410-SCU-080 consisting of one soil sample submitted for organochlorine herbicide analysis. The sample was analyzed by the S-Cubed laboratory using EPA method 8150. The sample identification number, collection date, and sample media are described in the following table.

SAMPLE ID	SAMPLE DATE	MEDIA
B07KP6	11/02/92	SOIL

Data validation was conducted in accordance with the WHC statement of work (WHC 1991) and validation procedures (Bechtold 1992). Attachments 1 through 4 to this memo provide the data validation supporting documentation and a summary of the validated results.

### DATA QUALITY OBJECTIVES

**Precision.** The laboratory did not analyze a matrix spike and matrix spike duplicate sample. Therefore, the relative percent differences could not be evaluated.

**Accuracy.** The laboratory did not analyze a matrix spike and matrix spike duplicate sample, therefore, percent recoveries (% R) could not be evaluated. The laboratory did analyze a laboratory control sample (LCS) in which the recoveries were evaluated and were acceptable.

The surrogate recovery was exceeded as noted in "Minor Deficiencies".

**Sample Result Verification.** All sample results were supported in the raw data with no data correction necessary.

**Detection Limits.** Detection limit goals were met.

**Completeness.** The data package was complete for all requested analyses. A total of one sample was validated in this data set with a total of 10 determinations reported. Out of the 10 determinations reported, all determinations were deemed valid which results in a completeness of 100 percent. This completeness percentage meets the work plan objectives of 90%.

### MAJOR DEFICIENCIES

There were no major deficiencies identified requiring rejection of the data.



## MINOR DEFICIENCIES

### Accuracy

The surrogate recovery exceeded the control limits for the method blank. Since this is a blank, no qualification of the sample data was necessary.

### Holding Times

The extraction holding time was exceeded for sample B07KP6. Therefore, the sample results were qualified as estimated (J for detects, UJ for non-detects).

## REFERENCES

WHC, 1993, Westinghouse Hanford Company, North Slope ERA Data Validation, Statement of Work, Revision 0, May 1993. Westinghouse Hanford Company, Richland, Washington.

Bechtold, 1992, Westinghouse Hanford Company, Data Validation Procedures for Chemical Analyses, WHC-SD-EN-SPP-002, Rev. 1, 1992. Westinghouse Hanford Company, Richland, Washington.

ATTACHMENT 1

GLOSSARY OF DATA REPORTING QUALIFIERS

## GLOSSARY OF ORGANIC DATA REPORTING QUALIFIERS

- B - Indicates the compound was analyzed for and detected in the associated blank. The "B" qualifier for organic data is applied by the laboratory only and is not applied by the data validators.
- U - Indicates the compound was analyzed for and not detected. The value reported is the sample quantitation limit corrected for sample dilution and moisture content by the laboratory. The data are usable for decision making purposes.
- UJ - Indicates the compound or analyte was analyzed for and not detected. Due to a quality control deficiency identified during data validation the value reported may not accurately reflect the sample quantitation limit. The data are usable for decision making purposes.
- J - Indicates the compound or analyte was analyzed for and detected. The associated value is estimated due to a quality control deficiency identified during data validation. The data are usable for decision making purposes.
- UR - Indicates the compound was analyzed for and not detected; however, due to an identified quality control deficiency the data are unusable.
- R - Indicates the compound was analyzed for and detected; however, due to an identified quality control deficiency the data are unusable.
- NJ - Indicates presumptive evidence of a compound at an estimated value.
- N - Indicates presumptive evidence of a compound.

ATTACHMENT 2  
SUMMARY OF DATA QUALIFICATIONS



ATTACHMENT 3  
AS QUALIFIED DATA SUMMARY

1D

**B07KP6**

**Contract:**

32359-51

SDG No.: 3410

Lab Code: S3

Case No.: 92-321

SAS No.:

SDG No.: 3410

Matrix: (soil/water) SOIL

Lab Sample ID: 3410-01

Sample wt/vol: 5 (g/ml) G

Lab File ID: H1120-4DB1701077

%Moisture: 5.67      decanted: (Y/N) N

Date Received: 11/05/92

Extraction: (SepF/Cont/Sonc) SEPF

Date Extracted: 11/16/92

Concentrated Extract Volume: 5000 (uL)

Date Analyzed: 11/22/92

Injection Volume: 1.00 (uL)

Dilution Factor: 1.00  
Sulfur Chlorine: (N/N)

GPC Cleanup: (Y/N) N pH: 8.95

Sulfur Cleanup: (Y/N) N

CONCENTRATION UNITS:

(ug/L or ug/Kg) ug/kg

Q

Q  
 uJ  
 uJ  
 uJ  
 uJ  
 uJ  
 uJ  
 uJ  
 uJ  
 uJ  
 uJ

5/6/93

0004

ATTACHMENT 4  
DATA VALIDATION SUPPORTING DOCUMENTATION



*Organochlorine* HERBICIDE DATA VALIDATION CHECKLIST - FORM A-4

PROJECT: <i>North Slope ERH</i>	REVIEWER: <i>G</i>	DATE: <i>6/9/93</i>
LABORATORY: <i>S - Cured</i>	CASE: <i>92-321</i>	SDG: <i>3410</i>
SAMPLES/MATRIX: <i>Soil BOKP6</i>		

## 1. DATA PACKAGE COMPLETENESS

Review the data package for completeness and check off the items below. If any data review elements are missing contact the laboratory for submittal.

<u>Data Package Item</u>	<u>Present?:</u>	<u>Yes</u>	<u>No</u>	<u>N/A</u>
Case Narrative		<input checked="" type="checkbox"/>		
Data Summary			<input checked="" type="checkbox"/>	
Chain of Custody Forms		<input checked="" type="checkbox"/>		
Sample Analysis Request		<input checked="" type="checkbox"/>		
QC Summary		<input checked="" type="checkbox"/>		
Surrogate Recovery		<input checked="" type="checkbox"/>		
MS/MSD Recovery			<input checked="" type="checkbox"/>	<i>See comment 1</i>
Method Blank Summary		<input checked="" type="checkbox"/>		
Sample Data				
Sample Results		<input checked="" type="checkbox"/>		
Chromatograms for all samples/extracts		<input checked="" type="checkbox"/>		
Quantitation sheets for all samples/extracts		<input checked="" type="checkbox"/>		
Extraction data sheets for all samples/extracts		<input checked="" type="checkbox"/>		
Instrument time/run logs for all samples/extracts		<input checked="" type="checkbox"/>		
Standards Data				
Initial Calibration standard concentrations		<input checked="" type="checkbox"/>		
Initial Calibration summary of RRF/RSD data		<input checked="" type="checkbox"/>		
Chromatograms for all initial cal. standards		<input checked="" type="checkbox"/>		
Quantitation sheets for all initial cal. standards		<input checked="" type="checkbox"/>		
Instrument time/run logs for all samples/extracts		<input checked="" type="checkbox"/>		
Calibration standard traceability data		<input checked="" type="checkbox"/>		
Raw QC Data				
Blanks				
Laboratory Blank results		<input checked="" type="checkbox"/>		
Chromatograms for all laboratory blanks		<input checked="" type="checkbox"/>		
Quantitation reports for all laboratory blanks		<input checked="" type="checkbox"/>		
Matrix Spike/Matrix Spike Duplicates				
<del>LC/MS/MSD</del> Results		<input checked="" type="checkbox"/>		
Chromatograms		<input checked="" type="checkbox"/>		
5/6/9/98 Quantitation reports		<input checked="" type="checkbox"/>		

<u>Data Package Item</u>	<u>Present?:</u>	Yes	No	N/A
Additional Data				
Moisture/% Solids data sheets		✓	—	—
Calculation formulae		—	✓	—
Instrument Run/Time Logs		—	✓	—
Chemist notebook pages		—	✓	—
Sample preparation sheets		✓	—	—

## 2. HOLDING TIMES

Were all samples extracted within holding times? Yes No N/A

Were all samples analyzed within holding times? Yes No N/A

ACTION: If the extraction or analytical holding times were exceeded, but not by a factor of two, qualify all affected results as estimated (J for detects and UJ for nondetects). Otherwise, reject all nondetects (R) and qualify all detects as estimated (J).

## 3. INSTRUMENT CALIBRATION

## 3.1 INITIAL CALIBRATION

Was an initial calibration conducted prior to sample analysis? Yes No N/A

Are all RSD values <20%? Yes No N/A

ACTION: If the RSD criteria were not met, qualify all results as estimated (J for detects and UJ for nondetects).

## 3.2 CONTINUING CALIBRATION

Have continuing calibrations been conducted at the proper frequency? Yes No N/A

Are the ~~RRFs~~ <sup>RPDs < 15% 5/6/943 for DB1701 column only</sup> within  $\pm 15\%$  of the initial calibration average RF? Yes No N/A

Are the RT values for the calibration compounds within the retention time windows? Yes No N/A

ACTION: If the percent difference criteria or retention time windows are not met, qualify all associated data as estimated (J for detects, UJ for nondetects).

## 4. BLANKS

## 4.1 LABORATORY BLANKS

Has the laboratory analyzed at least one method blank per matrix in the sample batch? Yes No N/A

Are target compounds present in the laboratory blanks?

(Yes) No N/A

ACTION: Qualify all detected results in the samples that are < 5 times the amount in any laboratory blank as nondetects (U).

#### 4.2 FIELD BLANKS

Are target compounds present in the field blanks?

Yes No (N/A)

ACTION: Qualify all detected results in the samples that are < 5 times the amount in any valid field blank as nondetects (U).

### 5. ACCURACY

#### 5.1 SURROGATE RECOVERY

Are any surrogate recoveries out of specification?

See comment  
(Yes) No N/A

Are any surrogates nondetected?

Yes (No) N/A

ACTION: Surrogate recoveries out of specification will require qualification of all associated data as estimated (J for detects and UJ for nondetects). Surrogate recoveries that are 0% will require qualification of all detects as estimated (J) and the rejection of all nondetects (R).

#### 5.2 MATRIX SPIKE RECOVERY

Has the laboratory conducted a <sup>LCS</sup>MS/MSD analysis per matrix for the sample group?

(Yes) No N/A

Are there calculation or transcription errors?

Yes (No) See comment 3  
N/A

Are MS recoveries within specification?

(Yes) No N/A

ACTION: If MS/MSD analyses have not been conducted contact the laboratory for clarification. Review the MS/MSD recoveries in conjunction with other QC data such as surrogate recoveries and note the results in the validation narrative. If MS/MSD recoveries are out of specification and sample concentration is > 5 times the spike concentration, no qualification is required, otherwise qualify positive results as estimated (J) in all samples if associated surrogates are also out of specification. The qualification shall only be done on samples of similar matrix as the MS/MSD samples. If it is determined from the review that only the spiked samples are affected by the low recoveries, qualify only the results for the spiked sample as described above. If it is determined from the review that out of specification MS/MSD recoveries are indicative of systematic problems in the laboratory such as sample preparation or sample-specific matrix interferences this must be noted in the validation narrative along with the potential affect on the sample results.

## 5.3 PERFORMANCE AUDIT SAMPLES

Are performance audit sample results within the acceptance limits?

Yes No

(N/A)

ACTION: Note the results of the performance audit samples in the validation narrative.

## 6. PRECISION

## 6.1 MATRIX SPIKE/MATRIX SPIKE DUPLICATES

Are there any calculation or transcription errors?

Yes No

(N/A)

Are the RPD values within specification?

Yes No

(N/A)

ACTION: Review the MS/MSD results in conjunction with other QC data such as field duplicates and not the results in the validation narrative. If MS/MSD RPD values are out of specification and sample results are  $> 5 \times \text{CRQL}$  qualify positive results as estimated (J). If it is determined from the review that out of specification MS/MSD results are indicative of systematic problems in the laboratory such as sample preparation or sample-specific matrix interferences this must be noted in the validation narrative along with the potential affect on the sample results.

## 6.2 FIELD DUPLICATES

Are the field duplicate RPDs acceptable?

Yes No

(N/A)

ACTION: Note the results of the field duplicate samples in the validation narrative.

## 6.3 FIELD SPLIT SAMPLES

Are the field split RPDs acceptable?

Yes No

(N/A)

ACTION: Note the results of the field split samples in the validation narrative.

## 7. COMPOUND IDENTIFICATION AND QUANTITATION

## 7.1 COMPOUND IDENTIFICATION

Are positive results within the retention time windows?

Yes No

(N/A)

Are positive results unaffected by interfering peaks?

Yes No

N/A

ACTION: If positive results are not within the retention time windows qualify all detected results as nondetects as follows: If the misidentified peak is outside the retention time windows and no potential interferences are present, report the CRQL and if the misidentified peak interferes with the potential detection of a target peak then the reported value is the quantitation limit and the result is qualified as estimated (UJ).

*See comment*

## 7.2 REPORTED RESULTS AND QUANTITATION LIMITS

Has the laboratory reported sample quantitation limits within 5xCRQL levels?

☒ Yes    No    N/A

Are there any calculation or transcription errors?

Yes    ☒ No    N/A

ACTION: If the results and quantitation limits are in error contact the laboratory for clarification and discuss in the validation narrative.

## 8. OVERALL ASSESSMENT AND SUMMARY

Has the laboratory conducted the analysis in accordance with the analytical SOW?

☒ Yes    No    N/A

Were project specific data quality objectives met for this analysis?

☒ Yes    No    N/A

ACTION: Summarize all the data qualifications and complete the data validation narrative as specified in Section 10.0 of the data validation requirements.

## COMMENTS (attach additional sheets as necessary):

1. The laboratory did not do an MS/MSD test and do an LCS that will be evaluated for recovery.
2. Surrogate recovery was above the control limit of 75-150 (181% and 190%) for the Blank and LCS. ~~Sample results~~ will not be qualified because of this.
3. LCS concentrations reported as slightly different than re-calculated values from the quantitation report, which may be due to rounding. All values are acceptable.
4. There were no positive identifications.

## HOLDING TIME SUMMARY - FORM B-1

[illegible]

## MEMORANDUM

TO: North Slope ERA Project QA Record

June 11, 1993

FR: Christina Jensen, Golder Associates Inc.

RE: Organophosphorus Pesticide Analysis Data Validation Summary for 3410-SCU-080

### INTRODUCTION

This memo presents the results of data validation on data package 3410-SCU-080 consisting of one soil sample submitted for organophosphorus pesticide analysis. The sample was analyzed by the S-Cubed laboratory using EPA method 8140. The sample identification number, collection date, and sample media are described in the following table.

SAMPLE ID	SAMPLE DATE	MEDIA
B07KP6	11/02/92	SOIL

Data validation was conducted in accordance with the WHC statement of work (WHC 1991) and validation procedures (Bechtold 1992). Attachments 1 through 4 to this memo provide the data validation supporting documentation and a summary of the validated results.

### DATA QUALITY OBJECTIVES

**Precision.** Goals for precision were met with the exception of the compounds in the individual mix A with the exception of sulprophos as noted in "Minor Deficiencies".

**Accuracy.** The laboratory analyzed a laboratory control sample (LCS) instead of a matrix spike (MS) and matrix spike duplicate (MSD) with results summarized in "Major Deficiencies".

Surrogate recoveries were also outside control limits as noted in "Minor Deficiencies".

**Sample Result Verification.** All sample results were supported in the raw data with no data correction necessary.

**Detection Limits.** Detection limit goals were met.

**Completeness.** The data package was complete for all requested analyses. A total of one sample was validated in this data set with a total of 21 determinations reported. Out of the 21 determinations reported, a total of 19 determinations were deemed valid which results in a completeness of 90 percent. This completeness percentage meets the work plan objectives of 90%.



## MAJOR DEFICIENCIES

The LCS recoveries were low for merphos (6.2%) and high for bolstar (2192%) and coumaphos (318%). Therefore, sample results were qualified as unusable (R for detects, UR for non-detects).

## MINOR DEFICIENCIES

### Calibrations

The initial calibration relative percent differences (%RPD) of 25% was exceeded for all compounds in the individual mix A with the exception of sulprophos. Therefore, the results for sample B07KP6 were qualified as estimated (J for detects, UJ for non-detects).

### Surrogates

The surrogate control limit of 40 - 140% was exceeded for sample B07KP6 (157%). Therefore, the results have been qualified as estimated (J for detects, UJ for non-detects).

### Matrix Spike/Matrix Spike Duplicates

An MS and MSD were not analyzed for this sample delivery group and therefore were not evaluated.

## REFERENCES

WHC, 1993, Westinghouse Hanford Company, North Slope ERA Data Validation, Statement of Work, Revision 0, May 1993. Westinghouse Hanford Company, Richland, Washington.

Bechtold, 1992, Westinghouse Hanford Company, Data Validation Procedures for Chemical Analyses, WHC-SD-EN-SPP-002, Rev. 1, 1992. Westinghouse Hanford Company, Richland, Washington.

ATTACHMENT 1  
GLOSSARY OF DATA REPORTING QUALIFIERS

## GLOSSARY OF ORGANIC DATA REPORTING QUALIFIERS

- B - Indicates the compound was analyzed for and detected in the associated blank. The "B" qualifier for organic data is applied by the laboratory only and is not applied by the data validators.
- U - Indicates the compound was analyzed for and not detected. The value reported is the sample quantitation limit corrected for sample dilution and moisture content by the laboratory. The data are usable for decision making purposes.
- UJ - Indicates the compound or analyte was analyzed for and not detected. Due to a quality control deficiency identified during data validation the value reported may not accurately reflect the sample quantitation limit. The data are usable for decision making purposes.
- J - Indicates the compound or analyte was analyzed for and detected. The associated value is estimated due to a quality control deficiency identified during data validation. The data are usable for decision making purposes.
- UR - Indicates the compound was analyzed for and not detected; however, due to an identified quality control deficiency the data are unusable.
- R - Indicates the compound was analyzed for and detected; however, due to an identified quality control deficiency the data are unusable.
- NJ - Indicates presumptive evidence of a compound at an estimated value.
- N - Indicates presumptive evidence of a compound.

ATTACHMENT 2  
SUMMARY OF DATA QUALIFICATIONS

**B-7**

ATTACHMENT 3  
AS QUALIFIED DATA SUMMARY

## EPA SAMPLE NO.

B07KP6

CAS NO.	COMPOUND	CONCENTRATION UNITS: (ug/L or ug/Kg) ug/kg	Q
---------	----------	---	---

115-90-2	Fensulfothion	53.0	U
13194-48-4	Ethoprop	21.2	U
150-50-5	Merphos	21.2	U
2921-88-2	Chlorpyrifos	21.2	U
298-00-0	Parathion-methyl	10.6	U
298-02-2	Phorate	10.6	U
298-04-4	Disulfoton	10.6	U
299-84-3	Ronnel	21.2	U
300-76-5	Naled	21.2	U
327-98-0	Trichloronate	21.2	U
333-41-5	Diazinon	21.2	U
34843-46-4	Tokuthion (Prothiofos)	10.6	U
35400-43-2	Bolstar (Sulprophos)	10.6	U
55-38-9	Fenthion	10.6	U
56-72-4	Coumaphos	53.0	U
62-73-7	Dichlorvos	10.6	U
7786-34-7	Mevinphos	42.4	U
8065-48-3	Dematon-O	42.4	U
8065-48-3A	Dematon-P	42.4	U
86-50-0	Azinphos methyl	53.0	U
961-11-5	Stirophos (Tetrachlorvinphos)	53.0	U

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ATTACHMENT 4

DATA VALIDATION SUPPORTING DOCUMENTATION

1



WHC-SD-EN-SPP-002, Rev. 1  
*Organophosphorus Pesticides*  
 HERBICIDE DATA VALIDATION CHECKLIST - FORM A-4

PROJECT: <i>North Slope ERA</i>	REVIEWER: <i>G</i>	DATE: <i>6/9/93</i>
LABORATORY: <i>S. Cubed</i>	CASE: <i>92-321</i>	SDG: <i>3410</i>
SAMPLES/MATRIX: <i>soils BOKP10</i>		

1. DATA PACKAGE COMPLETENESS

Review the data package for completeness and check off the items below. If any data review elements are missing contact the laboratory for submittal.

Data Package Item	Present?:	Yes	No	N/A
Case Narrative		✓		
Data Summary		✓	✓	
Chain of Custody Forms		✓		
Sample Analysis Request		✓		
QC Summary				
Surrogate Recovery		✓		
MS/MSD Recovery		✓	✓	<i>See comment</i>
Method Blank Summary		✓		
Sample Data				
Sample Results		✓		
Chromatograms for all samples/extracts		✓		
Quantitation sheets for all samples/extracts		✓		
Extraction data sheets for all samples/extracts		✓		
Instrument time/run logs for all samples/extracts				✓
Standards Data				
Initial Calibration standard concentrations		✓		
Initial Calibration summary of RRF/RSD data		✓		
Chromatograms for all initial cal. standards		✓		
Quantitation sheets for all initial cal. standards		✓		
Instrument time/run logs for all samples/extracts				✓
Calibration standard traceability data				✓
Raw QC Data				
Blanks				
Laboratory Blank results		✓		
Chromatograms for all laboratory blanks		✓		
Quantitation reports for all laboratory blanks		✓		
Matrix Spike/Matrix Spike Duplicates				
MS/MSD Results		✓		
Chromatograms		✓		
Quantitation reports		✓		

<u>Data Package Item</u>	<u>Present?:</u>	Yes	No	N/A
Additional Data				
Moisture/% Solids data sheets		✓	—	—
Calculation formulae		—	✓	—
Instrument Run/Time Logs		—	✓	—
Chemist notebook pages		—	✓	—
Sample preparation sheets		✓	—	—

## 2. HOLDING TIMES

Were all samples extracted within holding times? Yes No N/A

Were all samples analyzed within holding times? Yes No N/A

ACTION: If the extraction or analytical holding times were exceeded, but not by a factor of two, qualify all affected results as estimated (J for detects and UJ for nondetects). Otherwise, reject all nondetects (R) and qualify all detects as estimated (J).

## 3. INSTRUMENT CALIBRATION

### 3.1 INITIAL CALIBRATION

Was an initial calibration conducted prior to sample analysis? Yes No N/A

Are all RSD values <20%? Yes No N/A

ACTION: If the RSD criteria were not met, qualify all results as estimated (J for detects and UJ for nondetects).

### 3.2 CONTINUING CALIBRATION

Have continuing calibrations been conducted at the proper frequency?

Are the <sup>RPDs</sup> 25 within  $\pm 15\%$  of the initial calibration average RF? Yes No <sup>see comment 2</sup> N/A

Are the RT values for the calibration compounds within the retention time windows?

Yes No N/A

ACTION: If the percent difference criteria or retention time windows are not met, qualify all associated data as estimated (J for detects, UJ for nondetects).

## 4. BLANKS

### 4.1 LABORATORY BLANKS

Has the laboratory analyzed at least one method blank per matrix in the sample batch?

Yes No N/A

*See comment 3*

Are target compounds present in the laboratory blanks?

☒ Yes    No    N/A

ACTION: Qualify all detected results in the samples that are  $< 5$  times the amount in any laboratory blank as nondetects (U).

## 4.2 FIELD BLANKS

Are target compounds present in the field blanks?

Yes    No    ☒ N/A

ACTION: Qualify all detected results in the samples that are  $< 5$  times the amount in any valid field blank as nondetects (U).

## 5. ACCURACY

## 5.1 SURROGATE RECOVERY

Are any surrogate recoveries out of specification?

☒ Yes    No    N/A

Are any surrogates nondetected?

Yes    ☒ No    N/A

ACTION: Surrogate recoveries out of specification will require qualification of all associated data as estimated (J for detects and UJ for nondetects). Surrogate recoveries that are 0% will require qualification of all detects as estimated (J) and the rejection of all nondetects (R).

## 5.2 MATRIX SPIKE RECOVERY

*See 9/14/13*  
Has the laboratory conducted a MS/MSD analysis per matrix for the sample group?

*See comment 4*  
☒ Yes    No    N/A

Are there calculation or transcription errors?

Yes    ☒ No    N/A

Are MS recoveries within specification?

Yes    ☒ No    N/A

ACTION: If MS/MSD analyses have not been conducted contact the laboratory for clarification. Review the MS/MSD recoveries in conjunction with other QC data such as surrogate recoveries and note the results in the validation narrative. If MS/MSD recoveries are out of specification and sample concentration is  $> 5$  times the spike concentration, no qualification is required, otherwise qualify positive results as estimated (J) in all samples if associated surrogates are also out of specification. The qualification shall only be done on samples of similar matrix as the MS/MSD samples. If it is determined from the review that only the spiked samples are affected by the low recoveries, qualify only the results for the spiked sample as described above. If it is determined from the review that out of specification MS/MSD recoveries are indicative of systematic problems in the laboratory such as sample preparation or sample-specific matrix interferences this must be noted in the validation narrative along with the potential affect on the sample results.

## 5.3 PERFORMANCE AUDIT SAMPLES

Are performance audit sample results within the acceptance limits?

Yes No N/A

ACTION: Note the results of the performance audit samples in the validation narrative.

## 6. PRECISION

## 6.1 MATRIX SPIKE/MATRIX SPIKE DUPLICATES

Are there any calculation or transcription errors?

Yes No N/A

Are the RPD values within specification?

Yes No N/A

ACTION: Review the MS/MSD results in conjunction with other QC data such as field duplicates and not the results in the validation narrative. If MS/MSD RPD values are out of specification and sample results are  $> 5 \times \text{CRQL}$  qualify positive results as estimated (J). If it is determined from the review that out of specification MS/MSD results are indicative of systematic problems in the laboratory such as sample preparation or sample-specific matrix interferences this must be noted in the validation narrative along with the potential affect on the sample results.

## 6.2 FIELD DUPLICATES

Are the field duplicate RPDs acceptable?

Yes No N/A

ACTION: Note the results of the field duplicate samples in the validation narrative.

## 6.3 FIELD SPLIT SAMPLES

Are the field split RPDs acceptable?

Yes No N/A

ACTION: Note the results of the field split samples in the validation narrative.

## 7. COMPOUND IDENTIFICATION AND QUANTITATION

## 7.1 COMPOUND IDENTIFICATION

Are positive results within the retention time windows?

Yes No N/A

Are positive results unaffected by interfering peaks?

Yes No N/A

ACTION: If positive results are not within the retention time windows qualify all detected results as nondetects as follows: If the misidentified peak is outside the retention time windows and no potential interferences are present, report the CRQL and if the misidentified peak interferes with the potential detection of a target peak then the reported value is the quantitation limit and the result is qualified as estimated (UJ).

*7/6/9/19/3 : none detected*

## 7.2 REPORTED RESULTS AND QUANTITATION LIMITS

Has the laboratory reported sample quantitation limits within 5xCRQL levels?

☒ Yes    No    N/A

Are there any calculation or transcription errors?

Yes    ☒ No    N/A

ACTION: If the results and quantitation limits are in error contact the laboratory for clarification and discuss in the validation narrative.

## 8. OVERALL ASSESSMENT AND SUMMARY

Has the laboratory conducted the analysis in accordance with the analytical SOW?

☒ Yes    No    N/A

Were project specific data quality objectives met for this analysis?

☒ Yes    No    N/A

ACTION: Summarize all the data qualifications and complete the data validation narrative as specified in Section 10.0 of the data validation requirements.

COMMENTS (attach additional sheets as necessary):

1. Laboratory performed an LCS analysis instead of an 115/1157. % recoveries will be reviewed.
  2. The RPDs were outside ( $\geq 25$ ) limits for formaldehyde, ethoprop. all compounds in individual mix. As the results will be qualified as For (with the exception of sulfoxprop sulprophos)
  3. The surrogate recovery for the sample, B07K06 is 157% which is above the control limits of 40-140%. All results will be qualified as For or UT. Surrogate recovery for the LCS is also exceeded however this does not affect sample qualification.
  4. The LCS recoveries are outside QC limits of 40-140 as follows; with qualification as follows
- | Compound         | %R    | Qualification |
|------------------|-------|---------------|
| ethoprop         | 128   | none          |
| Meiphos          | 6.2   | R             |
| methyl parathion | 296.7 | none          |
| chlorate         | 140.9 | none          |
| Monnel           | 148.4 | none          |
| Diazinon         | 137.6 | none          |
| Malstar          | 219.2 | R             |
| disinaphos       | 318.2 | R             |
| methyl Azinophos | 188.5 | none          |

## HOLDING TIME SUMMARY - FORM B-1

[illegible]

## MEMORANDUM

TO: North Slope ERA Project QA Record

June 10, 1993

FR: Christina Jensen, Golder Associates Inc.

RE: General Chemistry Analysis Data Validation Summary for 3410-SCU-080

### INTRODUCTION

This memo presents the results of data validation on data package 3410-SCU-080 consisting of one soil sample submitted for anions, hexavalent chromium, and nitrate+nitrite as N. The sample was analyzed by the S-Cubed laboratory using routine laboratory protocols. The sample identification number, collection date, and sample media are described in the following table.

SAMPLE ID	SAMPLE DATE	MEDIA
B07KP6	11/02/92	SOIL

Data validation was conducted in accordance with the WHC statement of work (WHC 1993) and validation procedures (Bechtold 1992). Attachments 1 through 4 to this memo provide the data validation supporting documentation and a summary of the validated results.

### DATA QUALITY OBJECTIVES

**Precision.** Goals for precision were met.

**Accuracy.** Goals for accuracy were met with the exception of the matrix spike recovery for chloride as noted in "Minor Deficiencies".

**Sample Result Verification.** All sample results were supported in the raw data with no data correction necessary.

**Detection Limits.** Detection limit goals were met for all analyses.

**Completeness.** The data package was complete for all requested analyses. A total of one (1) sample was validated in this data set with a total of nine (9) determinations reported. Out of the nine (9) determinations reported, all determinations were deemed valid which results in a completeness of 100 percent. This completeness percentage meets the work plan objectives of 90%.

### MAJOR DEFICIENCIES

There were no major deficiencies identified during validation.



## MINOR DEFICIENCIES

### Holding Time

The holding time of 2 days was exceeded for ortho-phosphate; therefore, the sample result was qualified as estimated (J).

### Matrix Spike

The matrix spike recovery for chloride was 10.2%. Therefore, the sample result was qualified as estimated (J for detects, UJ for non-detects).

## REFERENCES

WHC, 1993, Westinghouse Hanford Company, North Slope ERA Data Validation, Statement of Work, Revision 0, May 1993. Westinghouse Hanford Company, Richland, Washington.

Bechtold, 1992, Westinghouse Hanford Company, Data Validation Procedures for Chemical Analyses, WHC-SD-EN-SPP-002, Rev. 1, 1992. Westinghouse Hanford Company, Richland, Washington.

ATTACHMENT 1  
GLOSSARY OF DATA REPORTING QUALIFIERS

## GLOSSARY OF INORGANIC DATA REPORTING QUALIFIERS

- B - Indicates the analyte was analyzed for and detected. The value reported is less than the contract required quantitation limit (CRQL) but greater than the instrument detection limit (IDL). The data are usable for decision making purposes.
- U - Indicates the analyte was analyzed for and not detected. The value reported is the sample quantitation limit corrected for sample dilution and moisture content by the laboratory. The data are usable for decision making purposes.
- UJ - Indicates the analyte was analyzed for and not detected. Due to a quality control deficiency identified during data validation the value reported may not accurately reflect the sample quantitation limit. The data are usable for decision making purposes.
- BJ - Indicates the analyte was analyzed for and detected at a concentration greater than the IDL but less than the CRQL. The associated value is estimated due to a deficiency identified during data validation. The data are usable for decision making purposes.
- J - Indicates the analyte was analyzed for and detected at a concentration greater than the CRQL. The associated value is estimated due to a deficiency identified during data validation. The data are usable for decision making purposes.
- UR - Indicates the analyte was analyzed for and not detected; however, due to an identified quality control deficiency the data are unusable.
- R - Indicates the analyte was analyzed and detected; however, due to an identified quality control deficiency the data are unusable.

ATTACHMENT 2  
SUMMARY OF DATA QUALIFICATIONS



ATTACHMENT 3  
AS QUALIFIED DATA SUMMARY

## ANIONS ANALYSIS

PAGE 1 OF 1

LABORATORY: S-CUBED  
 CLIENT: WHP  
 PROJECT: 92-359  
 LOT #: 3410  
 FILE #: ANI3410S  
 DISK #: ANI1123  
 METHOD NO.: 300.0  
 UNIT: MG/KG

DATA REVIEWER: *an 12/02/92*  
 PROJECT REVIEWER:  
 CHARGE #: 32359-51  
 DATE SAMPLED: 11-02-92  
 DATE RECEIVED: 11-05-92  
 PREP DATE: 11-09-92  
 DATE ANALYZED: 11-13-92  
 SAMPLE TYPE: SOIL

*R5/18*

LAB ID	P	Cl	NO2	Br	NO3	PO4	SO4	<i>Gr IV</i>
13410-01	1.96	10.9 <i>J</i>	<i>40.1</i> <i>cu</i> <i>&lt;0.2</i>	<i>40.1</i> <i>cu</i> <i>&lt;0.2</i>	13.0	1.43 <i>J</i>	<i>240.311</i> <i>1.8</i>	
13410-010 <i>cu</i>		<i>13.2</i> <i>cu</i>					<i>311</i> <i>cu</i>	

SO<sub>4</sub> result was required 10x dilution due to high concentration level. All other anions were reported on straight analysis run. All QC requirement were met. The sample was leached (20gm into 100ml) into DI type II water prior to analysis.

*aj6/10/93*

**S - CUBED**

# Trace Inorganics Report

Client: WHC  
Project: 92-231  
Sampling Date: 11/02/92

Analyst: EA  
Review: un 11/19  
Receipt. Date: 11/05/92

Analyte: CRVI

[illegible]

Method Detection Limit: 5.000 ug/L  
Preparation Method: SW7196  
Analytical Method: SW7196  
Preparation Date: 11/09/92  
Analysis Date: 11/11/92

UN = Units = (A=mg/kg B=ug/L C=mg/L) MT = Matrix = (S=Soil W=Water)

Comments: All QC requirement were excellent. The sample was required 1 to 5 dilution prior to analysis due to matrix interferences. The sample of 20 gm was cracked into 100 mL DI type II  $H_2O$  prior to analysis.





ATTACHMENT 4

DATA VALIDATION SUPPORTING DOCUMENTATION

## WET CHEMISTRY DATA VALIDATION CHECKLIST - FORM A-7

PROJECT: <i>North Slope ERA</i>	REVIEWER: <i>Cj</i>	DATE: <i>6/10/93</i>
LABORATORY: <i>S-Pubed</i>	CASE: <i>92-321</i>	SDG: <i>3410</i>
SAMPLES/MATRIX: <i>soil BotKPLc</i>		

## 1. DATA PACKAGE COMPLETENESS

Review the data package for completeness and check off the items below. If any data review elements are missing contact the laboratory for submittal of the omitted data.

<u>Data Package Item</u>	<u>Present?:</u>	<u>Yes</u>	<u>No</u>	<u>N/A</u>
Case Narrative		<input checked="" type="checkbox"/>		
Cover Page		<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>
Traffic Reports/Chain-of-Custody		<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Sample Analysis Data Report Forms		<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Standards Data		<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
QC Summary		<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Blanks Summary Report Forms		<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Spike Sample Recovery Report Forms		<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Duplicate Sample Analysis Report Forms		<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Laboratory Control Sample Report Forms		<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Raw Data		<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Ion Chromatograph Chromatograms		<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>
TOC and TOX Instrument Printouts		<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Laboratory Bench Sheets		<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Additional Data		<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Laboratory Sample Preparation Logs		<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Instrument Run Logs		<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Internal Laboratory Chain-of-Custody		<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>
Percent Solids Analysis Records		<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>
Reduction Formulae		<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>
Chemist Notebook Pages		<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>

## 2. HOLDING TIMES

Were all samples analyzed within holding times?

Yes ☐ No ☒ N/A

Action: If any holding times were exceeded qualify all affected results as estimated (J for detects and UJ for nondetects).

## 3. INITIAL CALIBRATIONS

Were all instruments calibrated daily, each set-up time and were the proper number of standards used?

☒ Yes    No    N/A

Are the correlation coefficients  $\geq 0.995$ ?

☒ Yes    No    N/A

Was a balance check conducted prior to the TDS analysis?

Yes    No    ☒ N/A

Was the titrant normality checked?

Yes    No    ☒ N/A

ACTION: Qualify all data as unusable (R) if reported from an analysis in which the above criteria were not met.

## 4. INITIAL AND CONTINUING CALIBRATION VERIFICATION

Have ICV and CCV been analyzed at the proper frequency?

☒ Yes    No    N/A

Are ICV and CCV percent recoveries within control?

☒ Yes    No    N/A

Are there calculation errors?

Yes    ☒ No    N/A

ACTION: Qualify all affected data in accordance with the validation requirements.

## 5. LABORATORY BLANKS

Are target analytes present in the laboratory blanks?

Yes    ☒ No    N/A

ACTION: Qualify all associated sample results for any analyte  $< 5$  times the amount in any laboratory blank as nondetected (U) and list the affected samples and analytes below.

## 6. FIELD BLANKS

Are target analytes present in the field blanks?

Yes    No    ☒ N/A

ACTION: Qualify all sample results for any analyte  $< 5$  times the amount in any valid field blank as nondetected (U).

## 7. MATRIX SPIKE SAMPLE ANALYSIS

Are spike recoveries within the acceptance limits?

☒ Yes    ☒ No    N/A

ACTION: If the sample concentration exceeds the spike concentration by a factor of 4 or more, and spike recoveries are outside the acceptance limits, no qualification is necessary. If spike recovery is outside the control limits and the sample results are  $> \text{CRQL}$ , qualify the data as estimated (J). If the spike recovery is  $< 30\%$  and the sample results are less than the IDL qualify the data as unusable (R).

4/26/10/4/28 see comment 1

## 8. LABORATORY CONTROL SAMPLE

Are percent recoveries within the acceptance limits?

☒ Yes    No    N/A

Are there calculation errors?

Yes    ☒ No    N/A

ACTION: Qualify the affected results according to the following requirements:

AQUEOUS LCS - Qualify as estimated (J), all sample results > IDL, for which the LCS %R falls within the range 50-79% or > 120%. Qualify as estimated (UJ), all sample results < IDL, for which the LCS falls within the range of 50-79%. Qualify as unusable (R) all sample results, for which the LCS %R < 50%.

SOLID LCS - Qualify as estimated (J), all sample results > IDL for which the LCS %R is outside the established control limits. Qualify as estimated (UJ), all sample results < IDL for which the LCS %R are lower than the established control limits.

## 9. PERFORMANCE AUDIT ANALYSES

Are the performance audit sample results within the acceptance limits?

Yes    No    ☒ N/A

ACTION: Note the results of the performance audit samples in the validation narrative.

## 10. DUPLICATE SAMPLE ANALYSIS

Are RPD values within the acceptance limits?

*See comment 2*  
☒ Yes    No    N/A

Action: Qualify the results for all associated samples of the same matrix as estimated (J) if the RPD falls outside the acceptance limits.

## 11. FIELD DUPLICATE SAMPLES

Do RPD values exceed the acceptance limits?

Yes    No    ☒ N/A

ACTION: Note the results of the field duplicate samples in the validation narrative.

## 12. FIELD SPLIT SAMPLES

Do RPD values exceed the acceptance limits?

Yes    No    ☒ N/A

ACTION: Note the results of the field split samples in the validation narrative.

### 13. ANALYTE QUANTITATION AND DETECTION LIMITS

Have results been reported and calculated correctly?

☒ Yes    No    N/A

Are instrument detection limits below the CRDL?

☒ Yes    No    N/A

Action: If analyte quantitation is in error, contact the laboratory for explanation. If errors or deficiencies can not be resolved with the laboratory, qualify associated data as unusable (R).

### 14. OVERALL ASSESSMENT AND SUMMARY

Has the laboratory conducted the analysis in accordance with the analytical SOW?

☒ Yes    No    N/A

Were project specific data quality objectives met for this analysis?

☒ Yes    No    N/A

ACTION: Summarize all the data qualifications and complete the data validation narrative as specified in Section 10.0 of the data validation requirements.

COMMENTS (attach additional sheets as necessary):

1. The MS recovery for chloride was 10.3% for sample B07KPC. The sample result will be qualified as estimated, Jc

2. The results for CrVI for B07KPC were  $< \text{MDL}$  for both sample and replicate. The RPD was reported as 30.9, however, the correct RPD is not calculable. Since the results were  $< \text{MDL}$  this RPD is OK.

## MEMORANDUM

TO: North Slope ERA Project QA Record

June 11, 1993

FR: Christina Jensen, Golder Associates Inc.

RE: Inorganic Analysis Data Validation Summary for 3410-SCU-080

### INTRODUCTION

This memo presents the results of data validation on data package 3410-SCU-080 consisting of one soil sample submitted for inorganics analysis. The sample was analyzed by the S-Cubed laboratory using CLP protocols. The sample identification, collection date and sample media are described in the following table.

SAMPLE ID	SAMPLE DATE	MEDIA
B07KP6	11/02/92	SOIL

Data validation was conducted in accordance with the WHC statement of work (WHC 1993) and validation procedures (Bechtold 1992). Attachments 1 through 4 to this memo provide the data validation supporting documentation and a summary of the validated results.

### DATA QUALITY OBJECTIVES

**Precision.** Goals for precision were met with the exception of selenium GFAA duplicate injection performance as noted in "Minor Deficiencies". The precision could not be evaluated based on matrix spike (MS) and matrix spike duplicate (MSD) results or laboratory duplicate results because the laboratory did not analyze these samples.

**Accuracy.** The laboratory did not analyze an MS or MSD. A laboratory control sample was analyzed and evaluated for accuracy with deficiencies noted in "Minor Deficiencies".

**Sample Result Verification.** All sample results were supported in the raw data with no data correction necessary.

**Detection Limits.** Detection limit goals were met for all analyses.

**Completeness.** The data package was complete for all requested analyses. A total of one sample was validated in this data set with a total of 23 determinations reported. Out of the 23 determinations reported, all determinations were deemed valid which results in a completeness of 100 percent. This completeness percentage meets the work plan objectives of 90%.

### MAJOR DEFICIENCIES

There were no major deficiencies identified during data validation.



## MINOR DEFICIENCIES

### GFAA Duplicate Injections

The relative standard deviation (%RSD) for selenium exceeded the QC limit of 20%. Therefore, the result for sample B07KP6 was qualified as estimated (J for detects, UJ for non-detects).

## REFERENCES

WHC, 1993, Westinghouse Hanford Company, North Slope ERA Data Validation, Statement of Work, Revision 0, May 1993. Westinghouse Hanford Company, Richland, Washington.

Bechtold, 1992, Westinghouse Hanford Company, Data Validation Procedures for Chemical Analyses, WHC-SD-EN-SPP-002, Rev. 1, 1992. Westinghouse Hanford Company, Richland, Washington.

ATTACHMENT 1  
GLOSSARY OF DATA REPORTING QUALIFIERS

## GLOSSARY OF INORGANIC DATA REPORTING QUALIFIERS

- B - Indicates the analyte was analyzed for and detected. The value reported is less than the contract required quantitation limit (CRQL) but greater than the instrument detection limit (IDL). The data are usable for decision making purposes.
- U - Indicates the analyte was analyzed for and not detected. The value reported is the sample quantitation limit corrected for sample dilution and moisture content by the laboratory. The data are usable for decision making purposes.
- UJ - Indicates the analyte was analyzed for and not detected. Due to a quality control deficiency identified during data validation the value reported may not accurately reflect the sample quantitation limit. The data are usable for decision making purposes.
- BJ - Indicates the analyte was analyzed for and detected at a concentration greater than the IDL but less than the CRQL. The associated value is estimated due to a deficiency identified during data validation. The data are usable for decision making purposes.
- J - Indicates the analyte was analyzed for and detected at a concentration greater than the CRQL. The associated value is estimated due to a deficiency identified during data validation. The data are usable for decision making purposes.
- UR - Indicates the analyte was analyzed for and not detected; however, due to an identified quality control deficiency the data are unusable.
- R - Indicates the analyte was analyzed and detected; however, due to an identified quality control deficiency the data are unusable.

ATTACHMENT 2  
SUMMARY OF DATA QUALIFICATIONS

**B-7**

ATTACHMENT 3  
AS QUALIFIED DATA SUMMARY

## U.S. EPA - CLP

1  
INORGANIC ANALYSES DATA SHEET

EPA SAMPLE NO.

3410-01

Lab Name: S\_CUBED Contract: 32359-51

Lab Code: S3 Case No.: 92231 SAS No.: SDG No.: 3410

Matrix (soil/water): SOIL Lab Sample ID: 3410-01

Level (low/med): LOW Date Received: 11/05/92

% Solids: 94.3

Concentration Units (ug/L or mg/kg dry weight): MG/KG

CAS No.	Analyte	Concentration	C	Q	M
7429-90-5	Aluminum	13400	-		P
7440-36-0	Antimony	12.7	U		P
7440-38-2	Arsenic	10.6	B		F
7440-39-3	Barium	157			P
7440-41-7	Beryllium	0.64	B		P
7440-43-9	Cadmium	0.64	B		P
7440-70-2	Calcium	16100	-		P
7440-47-3	Chromium	21.0	-		P
7440-48-4	Cobalt	14.6	-		P
7440-50-8	Copper	27.1	-		P
7439-89-6	Iron	27800	-		P
7439-92-1	Lead	29.9	-		F
7439-95-4	Magnesium	7700	-		P
7439-96-5	Manganese	571	-		P
7439-97-6	Mercury	0.11	U		CV
7440-02-0	Nickel	20.8	-		P
7440-09-7	Potassium	2330	-		P
7782-49-2	Selenium	6.4	U		F
7440-22-4	Silver	7.0	-		P
7440-23-5	Sodium	539	U		P
7440-28-0	Thallium	6.4	U		F
7440-62-2	Vanadium	52.2	-		P
7440-66-6	Zinc	96.1	-		P

Color Before: Clarity Before: Texture:

Color After: Clarity After: Artifacts:

Comments:

BO7KP6

ATTACHMENT 4  
DATA VALIDATION SUPPORTING DOCUMENTATION



## INORGANIC ANALYSIS DATA VALIDATION CHECKLIST - FORM A-6

PROJECT: North Slope FEA	REVIEWER: CJ	DATE: 6/5/98
LABORATORY: S - Cubed	CASE: 92-321	SDG: 3410
SAMPLES/MATRIX: Soil BULKY		

## 1. COMPLETENESS AND CONTRACT COMPLIANCE

Review the data package for completeness and check off the items below. If any data review elements are missing contact the laboratory for submittal of the omitted data.

Data Package Item	Present?:	Yes	No	N/A
Case Narrative		<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Cover Page		<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Traffic Reports		<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Sample Data		<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Inorganic Analysis Data Sheets		<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Standards Data		<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Initial and Continuing Calibration Verification		<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
CRDL Standard for AA and ICP		<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
QC Summary		<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Blanks		<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
ICP Interference Check Summary		<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Spike Sample Recovery		<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>
Post-Digestion Spike Sample Recovery		<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>
Duplicate		<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>
Laboratory Control Sample		<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Standard Addition Results		<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>
ICP Serial Dilutions		<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Instrument Detection Limits		<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
ICP Interelement Correction Factors		<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
ICP Linear Ranges		<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Preparation Log		<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Analysis Run Log		<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Raw Data		<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
ICP Raw Data		<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Furnace AA Raw Data		<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Mercury Raw Data		<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Cyanide Raw Data		<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>
Additional Data		<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>
Internal laboratory chain-of-custody		<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Laboratory Sample Preparation Records		<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>

<u>Data Package Item</u>	<u>Present?:</u>	<u>Yes</u>	<u>No</u>	<u>N/A</u>
Percent Solids Analysis Records		<u>—</u>	<u>✓</u>	<u>—</u>
Reduction Formulae		<u>—</u>	<u>✓</u>	<u>—</u>
Instrument Run Logs		<u>—</u>	<u>✓</u>	<u>—</u>
Chemist Notebook Pages		<u>—</u>	<u>✓</u>	<u>—</u>

**2. HOLDING TIMES**

Have all samples been analyzed within holding times? **Yes** No N/A

**ACTION:** If any holding times have been exceeded qualify all affected results as estimated (J for detects and UJ for nondetects).

**3. INITIAL CALIBRATIONS**

Were all instruments calibrated daily, each set-up time and were the proper number of standards used? **Yes** No N/A

Are the correlation coefficients  $\geq 0.995$ ? **Yes** No N/A

Was a midrange cyanide standard distilled? **Yes** No **N/A**

**ACTION:** Qualify all data as unusable if reported from an analysis in which an instrument was not calibrated or was calibrated with less than the minimum number of standards. Qualify associated sample results >IDL as estimated (J) and results <IDL as estimated (UJ), if the correlation coefficient is <0.995 or the laboratory did not distill the midrange cyanide standard.

**4. INITIAL AND CONTINUING CALIBRATION VERIFICATION**

Are ICV and CCV percent recoveries within control? **Yes** No N/A

Are there calculation errors? **Yes** **No** N/A

**ACTION:** Qualify all affected data in accordance with Section 8.3 of the validation requirements. If calculation errors are noted, contact the laboratory for clarification.

**5. ICP INTERFERENCE CHECK SAMPLE**

Has an ICS sample been analyzed at the proper frequency? **Yes** No N/A

Are the AB solution %R values within control? **Yes** No N/A

Are there calculation errors? **Yes** **No** N/A

**ACTION:** Qualify all affected data in accordance with Section 8.3 of the validation requirements. If calculation errors are noted, contact the laboratory for clarification.

## 6. LABORATORY BLANKS

Are target analytes present in the laboratory blanks?

See comment 1  
☒ Yes    No    N/A

**ACTION:** Qualify all associated sample results for any analyte <5 times the amount in any laboratory blank as nondetected (U). If analyte concentrations in the blank are > CRDL or below the negative CRDL, verify the laboratory has redigested and reanalyzed associated samples with analyte concentrations < 10 times the blank concentration. If the laboratory has not redigested and reanalyzed the samples, note in the validation narrative.

## 7. FIELD BLANKS

Are target analytes present in the field blanks?

Yes    No    ☒ N/A

**ACTION:** Qualify all sample results for any analyte <5 times the amount in any valid field blank as nondetected (U).

## 8. MATRIX SPIKE SAMPLE ANALYSIS

Are spike recoveries within the control limits?

Yes    No    ☒ N/A

**ACTION:** Qualify the affected sample data according to the following requirements:

If spike recovery is > 125% and sample results are <IDL no qualification is required. If spike recovery is > 125% or <75% qualify all positive results as estimated (J). If spike recovery is 30% to 74% qualify all nondetects as estimated (UJ). If spike recovery is <30%, reject all nondetects (R). If the field blank has been used for spike analysis, note in the validation narrative.

## 9. LABORATORY CONTROL SAMPLE

Are percent recoveries within the acceptance limits?

Yes    No    N/A

Are there calculation errors?

Yes    No    N/A

**ACTION:** Qualify the sample data according to the following requirements:

**AQUEOUS LCS** - Qualify as estimated (J), all sample results >IDL, for which the LCS %R falls within the range 50-79% or > 120%. Qualify as estimated (UJ), all sample results <IDL, for which the LCS falls within the range of 50-79%. Qualify as unusable (R) all sample results, for which the LCS %R <50%.

**SOLID LCS** - Qualify as estimated (J), all sample results >IDL for which the LCS result is outside the established control limits. Qualify as estimated (UJ), all sample results <IDL for which the LCS %R are lower than the established control limits.

10. PERFORMANCE AUDIT ANALYSES

Are the performance audit sample results within the acceptance limits?

Yes No N/A

ACTION: Note the results of the performance audit sample analyses in the data validation narrative.

11. DUPLICATE SAMPLE ANALYSIS

Are RPD values acceptable?

Yes No N/A

ACTION: Qualify the results for all associated samples of the same matrix as estimated (J) if the RPD results fall outside the appropriate control limits. If field blanks were used for laboratory duplicates, note in the validation narrative.

12. ICP SERIAL DILUTION

Are the serial dilution results acceptable?

Yes No N/A

Is there evidence of negative interference?

Yes No N/A

ACTION: Qualify the associated data as estimated (J) for those analytes in which the %D is outside the control limits. If evidence of negative interference is found, use professional judgment to qualify the data.

13. FIELD DUPLICATE SAMPLES

Do the RPD values exceed the control limits?

Yes No N/A

ACTION: Note the results of the field duplicate samples in the validation narrative.

14. FIELD SPLIT SAMPLES

Do the RPD values exceed the control limits?

Yes No N/A

ACTION: Note the results of the field split samples in the validation narrative.

1516. FURNACE ATOMIC ABSORPTION QUALITY CONTROL

Do all applicable analyses have duplicate injections?

Yes No N/A

Are applicable duplicate injection RSD values within control?

Yes No N/A

If no, were samples rerun once as required?

Yes No N/A

Does the RSD for the rerun fall within the control limits?

Yes No N/A

Were analytical spike recoveries within the control limits?

Yes No N/A

If no, were MSA analyses performed when required?

Yes No ☒ N/A

Are MSA correlation coefficients  $\geq 0.995$ ?

Yes No ☒ N/A

If no, was a second MSA analysis performed?

Yes No ☒ N/A

**ACTION:** If duplicate injections are outside the acceptance limits and the sample has not been reanalyzed or the reanalysis is outside the acceptance limits, qualify the associated data as estimated (J for detects and UJ for nondetects). If the analytical spike recovery is  $< 40\%$  qualify detects as estimated (J). If the analytical spike recovery is  $\geq 10\%$  but  $< 40\%$ , qualify all nondetects as estimated (UJ) and if the analytical spike recovery is  $< 10\%$ , reject all nondetects (R). If the sample absorbance is  $< 50\%$  of the analytical spike absorbance and the analytical spike recovery is  $< 85\%$  or  $> 115\%$ , qualify all results as estimated (J for detects and UJ for nondetects). If method of standard additions (MSA) was required but was not performed, the MSA samples were spiked incorrectly, or the MSA correlation coefficient was  $< 0.995$ , qualify the associated detected results as estimated (J).

## 17. ANALYTE QUANTITATION AND DETECTION LIMITS

Have results been reported and calculated correctly?

☒ Yes No N/A

Are results within the calibrated range of the instruments and within the linear range of the ICP?

☒ Yes No N/A

Are all detection limits below the CRQL?

☒ Yes No N/A

**Action:** If analyte quantitation is in error, contact the laboratory for explanation. If errors or deficiencies can not be resolved with the laboratory, qualify associated data as unusable (R).

## 18. OVERALL ASSESSMENT AND SUMMARY

Has the laboratory conducted the analysis in accordance with the analytical SOW?

☒ Yes No N/A

Were project specific data quality objectives met for this analysis?

☒ Yes No N/A

**ACTION:** Summarize all the data qualifications and complete the data validation narrative as specified in Section 10.0 of the data validation requirements.

## COMMENTS (attach additional sheets as necessary):

1. Lead and arsenic were detected in the lab prep. blank at .7 and 1.58 mg/kg. No data qualification was done on associated samples because the sample results are  $< 5 \times$  the blank values.
2. The laboratory did not analyze an INS/MSD. The LCS will be reviewed.
3. The LCS %R was 139.5 (limits 80-120) for Selenium. However, the selenium value is  $< 15\%$  so no qualification is necessary.
4. The laboratory did not perform a duplicate analysis. No qualification was done because of this.

## HOLDING TIME SUMMARY - FORM B-1

[illegible]

## BLANK AND SAMPLE DATA SUMMARY - FORM B-3

[illegible]